Industrial Waste Diversion Program

THE REDUCTION OF LEAD IN STEEL FLUE DUST USING A BATCH LEACHING PROCESS

JUNE 1991

Ontario
Environment
Environnement

ISBN 0-7729-8429-8

INDUSTRIAL WASTE DIVERSION PROGRAM

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Report prepared for :

Waste Management Branch
Ontario Ministry of the Environment

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PIBS 1578

THE REDUCTION OF LEAD IN STEEL FLUE DUST USING A BATCH LEACHING PROCESS

Report prepared for:

Waste Management Branch
Ontario Ministry of the Environment

In consultation with

University of Toronto

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92-00121/

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Abstract

The reduction of the concentration of lead present in a sample of steel flue dust was investigated in order to meet the Ministry of the Environment leachate requirements as stated in Regulation 309. The alkalinity of the leaching solution, liquid to solid ratio (L/S) of the loading, and temperature were varied. This was accomplished with a batch leaching process that lasted for a 24 hour period.

The leaching solutions used for the first experiments were deionized water, 0.47M NaOH, 0.96M NaOH, and 3.47M NaOH. The L/S ratio was constant at 167:1. As the alkalinity of the leaching solution increased, the amount of lead leached from the steel flue dust also increased. Approximately 62 ppm of lead was leached with the deionized water while 93 ppm was leached with the 3.47M NaOH. Also, the percent recovery of the total lead in the sample increased from 30% to 46% as the alkalinity increased.

The L/S ratios ranged from 3.3:1 to 167:1 for the second experiments. The leaching solution used was 0.47M KOH. As the L/S ratio decreased, the concentration of lead leached from the sample increased. The range of concentrations was from 1462 ppm to 81 ppm. However, as the L/S ratio decreased, the percent recovery of the available lead from the sample also decreased. At a 20:1 ratio, only 75% of the available lead was recovered and the percent dropped to about 40% for the 3.3:1 ratio.

The temperature ranged from 289 K to 353 K for the third experiments. For

each experiment, the L/S ratio was 167:1 and the leaching solution was 0.47M KOH. Little variation in the data was seen implying that temperature had negligible effects on the amount of lead leached from the steel flue dust at this concentration.

The MOE leach test was conducted on a treated sample from the 5:1 experiment. The average concentration was about 41 ppm or 1.5% of the available lead. The MOE limit was 5 ppm. Although it failed the test, there was a substantial decrease. Originally, 1325 ppm leached from an untreated sample or 49% of the available lead. It is hypothesized that any L/S ratio greater than 100:1 would pass the MOE test.

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1.0 INTRODUCTION

This report covers the initial investigation of the leaching characteristics of lead from steel flue dust. The key objective of this part of the investigation is to examine the possibility of reducing the concentration of lead present in the steel flue dust in order to meet the Ministry of the Environment (MOE) leachate requirements. The achievement of this goal would provide an opportunity to utilize the residues rather than having to use secure waste landfill site disposal.

Legislation requires that generators of waste register their waste if it meets one or more of the hazardous characteristics described in the MOE Waste Identification Flowchart [1]. The section of the Flowchart that pertains to this study is the leachate quality.

A waste cannot generate contaminants at concentrations greater than 100 times the concentrations listed in Schedule 4 of Regulation 309 [2]. Schedule 4 of the guideline states that the levels of lead in the leachate cannot exceed 100 times 0.05 mg/L or 5 parts per million (ppm). The MOE tests for contaminants using a standardized leach test [3]. If contaminants do leach out in concentrations that exceed the limits, then the waste is classified as hazardous.

The iron and steel industry generates considerable tonnages of solid waste each year. Although a large extent is recovered or recycled, some 50 kg of waste to every 1000 kg of steel have to be discharged or disposed of as waste [4]. Of that 50 kg, approximately 13 to 18 kg/tonne is steel flue dust [5]. With the strict guidelines imposed on waste to qualify for landfill dumping, the disposal of the steel flue dust in its present state presents a problem to the steel industry. Thus, a process is needed to treat the steel flue dust.

The reduction of the lead concentration present in the steel flue dust can be accomplished with a leaching process. Ideally, leaching yields a residue sufficiently free of the desired metal for it to be disposed and a metal-rich or pregnant solution from which the metals can be separated and recovered.

The method of leaching chosen was a batch process with a tumbling apparatus (refer to Figure 4.1). The parameters pH, loading (ie., liquid to solid mass ratio), and temperature were examined to determine the effects on the concentration of lead leached from the steel flue dust in a 24 hour period. Also, some long term experiments and the MOE leachate test were conducted.

Earlier work conducted by J.Kramer provides valuable information regarding the composition, mineralogy, and morphological nature of a typical sample of steel flue dust[6]. The results of the Atomic Absorption Spectroscopy (AAS) analysis indicates that lead is present at a concentration of 34000 mg/kg of sample [6]. Most of this lead is present as simple oxides or associated with Fe/Mn oxy-hydroxides.

2.0 LITERATURE REVIEW

2.1 American Leaching Tests

The governing bodies regulating batch laboratory leaching and extraction procedures are the American Society for Testing and Materials (ASTM) and the Environmental Protection Agency (EPA). Batch tests are differentiated from column tests based on the manner in which the extracting solutions contact the waste. Methods where the waste is continuously contacted with the same solution in a reaction vessel are classified as batch tests. Methods which utilize the continuous or intermittent introduction of fresh extractant through a column packed with waste are classified as column tests [7].

An extensive research was conducted by Perket and Webster outlining the major chronological events in the development of a standard test method [7]. Their major findings are outlined below. Early work developed in the early 1970s referenced the batch test adopted by the Japanese government in 1973. The Japanese procedure involved a continuously agitated solution at a pH between 5.8 and 6.3, maintained in contact with the waste at a 10:1 liquid to solid ratio for a period of 6 hours. The pH was adjusted by the addition of hydrochloric acid, carbon dioxide, or sodium hydroxide. The resulting solution was separated by filtration, and centrifuged before being analyzed. That same year, the EPA adapted a similar batch procedure referred to as the Elutriate Test.

Among the first published papers utilizing any of the batch tests on waste materials were those by Shannon in 1974 and by Weir in 1975. Shannon studied the solubilities of selected cations from lignite fly ashes by altering the water to waste ratio while Weir's studies examined pH and time effects.

The first attempt at developing a procedure to be utilized as a standard in evaluating leaching of wastes was designated the Standard Leach Test (SLT). The SLT utilized a 0.45 micron filter to separate the solid fraction, which was then mixed with the leaching medium in a 10:1 liquid to solid ratio and allowed to rotate for 24 hours. The leaching solution was either water or a buffered solution containing glycine, pyrogallol and ferrous sulphate.

By the late 1970s, the EPA proposed the Toxicant Extraction Procedure (TEP). The TEP was directed to the development of a method for classifying wastes as hazardous or nonhazardous based on their leaching potential under standard conditions. After deliberations and modifications, the modified version of the TEP was designated as the Extraction Procedure (EP).

The EP involved maintaining the extraction solution at a pH of 5.0 using a quantity of 0.5 N acetic acid solution, not exceeding 4 ml/g of waste, after which the pH was left alone. The liquid to solid ratio was 20:1. The extraction was done on an agitation or stirring device for a period of 24 hours.

At the same time, the ASTM was developing the ASTM test as an alternate method for use in evaluating wastes. ASTM developed two methods; the difference between the two being the use of a buffered acetic acid solution in one and a neutral water solution in the other. Neutral water was selected as a medium for further development because it offered less inherent interference with use of the extraction solution for biological testing, and is less biased in its ability to

solubilize some metals more than others. This method known as ASTM Method A was approved as a standard method and published in the 1982 Annual Book of ASTM Standards.

2.2 Canadian Leaching Tests

The Ontario guidelines for batch leach tests are governed by Regulation 309 [1]. It specifies a leachate extraction procedure (LEP) by which extraction is carried out at a pH near 5 which is achieved by successive adjustments of the waste slurry with acetic acid [3]. If concentrations exceed 100 times those in Schedule 4 of Regulation 309, then the solid is considered a toxic waste and hazardous [2].

An alternate method proposed is the five step sequential extraction procedure proposed by Tessier [8]. The first two steps are the most important. In the first step, a 1 gram sample is extracted at room temperature for 1 hour with 8 mL of 1 M $MgCl_2$ at pH of 7 and continuous agitation. In the second step, 8 mL of 1 M NaOAc, adjusted to a pH of 5 with acetic acid, is added to the residue from the above step. In both steps, the samples are centrifuged and the supernatant is removed for analysis of trace metals by AAS.

Depending upon the nature of the substrate and the binding of the trace metals, the trace metals may be mobile/immobile under acid/base, oxidizing/reducing, and varying solute chemistry [9].

A comparison of the two methods was conducted in a study by Kramer [10]. In all but one sample type, the two step sequential extraction mobilized significantly more trace metals than did the LEP. The differences were greatest for industrial chemical wastes and dusts.

The reason for the lower metal values for the LEP method are hypothesized by Kramer to be due to readsorption on substrates, especially iron/manganese oxides. If this hypothesis for the differences in mobility is true, the pH adjustment and regulation is the sensitive step in metal mobility.

Repeatability of results is a very important aspect when dealing with waste that may not have uniform composition. Kramer used samples exhibiting both large and small concentrations in replicate analyses. He found that repeatability of the tests was from 1-20% for metals in elevated concentrations (>100mg/kg) and from 10-100% for trace levels (<100mg/kg).

2.3 Analysis of Steel Flue Dust

A study was done by Kramer examining the mobilization of potentially toxic trace metals in industrial wastes [6]. Five industrial waste samples were obtained from the Ontario MOE for characterization. One of the samples was steel flue dust. The analyses of the samples involved various analytical techniques and methods.

Bulk chemistry analysis indicated that the steel flue dust was mainly Fe-Mn oxy-hydroxides with lesser Al and Si. Also, elevated lead and zinc were probably associated with Fe-Mn oxy-hydroxides or as simple oxides [11].

The mineral composition of the steel flue dust was determined by x-ray diffraction. This technique revealed that the sample was mostly magnetite with lesser amounts of hematite. The lead minerals were present as stable ferrites, silicates, and as metals [12]. A third technique used was sequential chemical extraction. The results from this analysis reiterated the fact that the majority of the

trace metals seem to be associated with strongly bound insoluble silicates and ferrites.

In this article, Kramer provided some valuable information regarding the composition, mineralogy, and morphological nature of steel flue dust. Of particular importance was the knowledge that lead existed as lead oxides and that 34000 mg of lead per kg of sample was
extracted or 3.4% lead by weight [6].

Similar studies conducted on electric arc furnace dust from Sweden and the United Kingdom confirmed Kramer's studies. The range of lead found in 14 samples was between 1.1% to 5.0% by weight [13].

2.4 Factors Affecting Leachability

2.4.1 Leaching Medium and L/S Ratio on Leachate pH

In a study performed by Cote and Constable, a number of wastes from various locations across Canada were subjected to a set of leaching tests [14]. Different leaching medias and liquid to solid ratios were used to examine the effects on leachate pH.

The results indicated that when distilled water was used as the leaching medium, the final pH of the leachate was controlled by the alkalinity of the waste. Also, changing the liquid to solid ratio had little effect on the leaching pH [15].

When an acidic solution was used as the leaching medium, the final leachate pH appeared to be a function of the interaction between the waste pH and the amount of acid available to react with the waste. Increasing the liquid to solid ratio increased the amount of acid contacting the waste and shifted the leachate pH towards the pH of the leaching medium [16].

The leachability of selected pollutants under various test conditions was also investigated. Of importance was the conclusion that for lead, a strong linear relationship existed between the leachate pH and the logarithm of lead released [17].

2.4.2 Liquid to Solid Ratio

In a study performed by the International Iron and Steel Institute (IISI), it was found that the liquid to solid ratio can affect test results in different ways depending on the elements involved. For lead and zinc, it was found that the rate of extraction was proportional to the liquid to solid ratio [18].

2.4.3 Stirring Time and Stirring Conditions

The time used by most of the standard test methods is 24 hours. This duration allows sufficient time for equilibrium to be achieved at the end of leaching. The IISI studied the effects of stirring time on the concentrations of chromium. The impact of stirring time was significant only for durations of less than a few hours. Beyond that, a balance was maintained [19].

The intensity with which stirring was done and how it was done can have a determinant effect on test results when the conditions of transfer at the solid/liquid interface control dissolution [19]. The IISI conducted slow 24 hour stirring and accelerated 3 hour stirring tests. They found that accelerated stirring favoured dissolution of cadmium and lead while slow stirring favoured chromium leaching

[20]. In a separate article reviewing these findings, the authors indicated that the ideal stirring process would be one which homogenized the liquid and renewed it at the solid/liquid interface, without altering the physical state of the waste material [19]. However, they concluded that in the absence of further information, that the impact of stirring was a nondiscriminating factor.

3.0 THEORETICAL BACKGROUND

Leaching is the process of dissolution of the value mineral of an ore or concentrate, generally by an aqueous solution of the leaching agent [21]. The term may be extended to include the dissolution of secondary materials such as scrap, residues, and wastes.

Ideally, leaching yields a residue sufficiently free of the desired minerals for it to be disposed and a metal-rich solution from which the metals can be separated and recovered. Leaching is always conducted with aqueous solutions in which there is a solubility of the dissolving minerals or compounds in the material being leached sufficient to obtain an acceptable concentration of valuable metals in solution [22].

Pourbaix established equations representing the influence of pH on the solubility of PbO and $Pb(OH)_2$. He found that plumbous oxide dissolved in very alkaline solutions as biplumbite ions having a minimum solubility at a pH of 9.34 (0.222 mg Pb/L) [23]. At pH values larger than 9.34, the solubility of lead increased.

4.0 EXPERIMENTAL SECTION

4.1 Equipment

All experiments were conducted in room 333 in the Walberg Building of the University of Toronto. The main apparatus used for the leaching experiments was a tumbling mixer. It consisted of a wooden box that was rotated by a small motor at a speed of 38 rpm. Three plastic containers used to contain the waste and the leaching solution, fit inside the box.

Sample analysis was performed using atomic absorption. The unit was located in room 233 in the Walberg Building. The model used was a Perkin-Elmer 703 Atomic Absorption Spectrophotometer.

Glassware needed for the experiments consisted of various sizes of beakers, erlenmeyer flasks, volumetric flasks, pipettes, and a burette. Samples that needed to be analyzed by AA were stored in 15 mL plastic vials. The filtering device consisted of a syringe and 0.45 micron filters. In addition to the above, a standard pH meter, a thermometer, and magnetic stirrers were also needed.

4.2 Materials

The steel flue dust was already present in the lab. The sample was collected prior to any experimentation circa 1985. Deionized water was used for every aspect of the experimental work. City tap water was only used for washing purposes.

The chemical reagents required were analytical grade sodium hydroxide, potassium hydroxide, potassium hydrogen phthalate, phenolphthalein, and

concentrated nitric acid. In order to prepare the calibration curve for the AAS, a solid compound containing lead (eg. lead acetate) was also needed.

4.3 Procedures

4.3.1 Standardization of Base Solutions

Prior to performing any standardizations, the potassium hydrogen phthalate was heated in an oven at 373 K for 1 hour to remove any moisture contained in the solid. The procedure involved dissolving approximately 0.5 g of the phthalate solid with about 50 mL of deionized water. A few drops of the phenolphthalein indicator was added and then the base of unknown concentration was added until a colour change (clear to pink) was noticed. The volume of the base required to produce the colour change was recorded. The titration was repeated three times.

At the end of each experiment, the above procedure was repeated. The purpose was to determine the effective hydroxide ion concentration and convert that concentration into a pH value.

4.3.2 Experimental Methods

4.3.2.1 Variation of pH

The following steps were carried out:

1. Weighed approximately 6 g of the steel flue dust.
2. Repeated step 1 two additional times.
3. Placed each 6 g sample in a 1 L plastic container.
4. Added 1 L of deionized water at room temperature.
5. Commenced the tumbling mixer for a duration of 24 hours.
6. Obtained 2 mL from each of the three samples at various times throughout the 24 hour period.
7. Filtered the slurry sample through a 0.45 micron filter paper.
8. Prepared the samples for AA analysis. Diluted the samples and added 1% by volume concentrated nitric acid.
9. At the conclusion of the experiment, 10 mL of the leaching solution was filtered and diluted by a factor of 10. The hydroxide ion concentration was determined.

The leaching solution used in step 4 varied according to the desired pH range being investigated. The solutions that were used were 0.47 M NaOH, 0.97 M NaOH, and 3.47 M NaOH.

4.3.2.2 Variation of Liquid to Solid Ratio

The above procedure was followed except for steps 1 and 4. The mass used in step 1 varied with the desired ratio. The ratios investigated were 167:1, 20:1, 10:1, 5:1, and 3.3:1. In each case, the leaching solution in step 4 was 0.47 M KOH.

4.3.2.3 Variation of Temperature

The temperatures investigated were 315, 327, 338, and 353 K. This was accomplished using a magnetic stirrer and hot plate. The liquid to solid ratio used was 167:1 (500 mL of solution with 3 g of sample) and the leaching solution was 0.47 M KOH. The duration of each run was 2 hours and 10 samples were taken

during that period.

5.0 RESULTS

The experimental results for the L/S ratio and temperature experiments are summarized in Tables 5.1 and 5.2. The results for the experiments with different leaching solutions as well as all the raw data is found in Appendix IV. Although the concentrations are reported in ppm, the units mg/L can also be used.

Table 5.1
Average Concentrations for Various L/S Ratios

	Average Lead Concentration (ppm)				
Time (hr)	167:1	20:1	10:1	5:1	3.3:1
0	0	0	0	0	0
0.083	81	503	870	1396	1589
0.333	82	479	944	1408	1534
0.833	80	503	893	1396	1501
2	78	495	895	1368	1469
4	83	490	868	1306	1420
7	-	473	866	1260	1410
24	85	455	890	1139	1309
average:	81	485	889	1325	1462

Table 5.2
Average Concentrations for Various Temperatures

	Average Lead Concentration (ppm) for Various Temperatures (°)			
Time (min)	315	327	338	353
0	0	0	0	0
2	83	75	82	68
4	88	79	84	80
7	97	85	85	77
10	93	83	86	82
15	86	82	81	78
25	87	79	92	78
40	92	78	89	79
60	84	84	97	86
90	90	84	98	79
120	88	85	89	76
average:	89	81	88	78

Since the analysis of the samples was done by atomic absorption, a

calibration curve was constructed relating absorption to concentration. Refer to Appendix I for the data, the calibration curve, and a statistical analysis. Each time a sample was analyzed, five absorption values were obtained and the average absorption was converted into a concentration.

The results from the various pH and L/S ratio experiments were converted into percents relating the total lead in the sample and the total available leached lead. Calculations are given in Appendix II.

A Pourbaix diagram relating the theoretical solubility of lead in the alkaline pH range is contained in Appendix III. The reactions and formulas used to derive the diagram are also included.

The MOE Leachate Test was performed on a treated sample from the 5:1 ratio experiment. Two runs were performed and in both cases the maximum allowable amount of acid to reduce the pH was added after 15 minutes (200 mL). The results are given in Table 5.3.

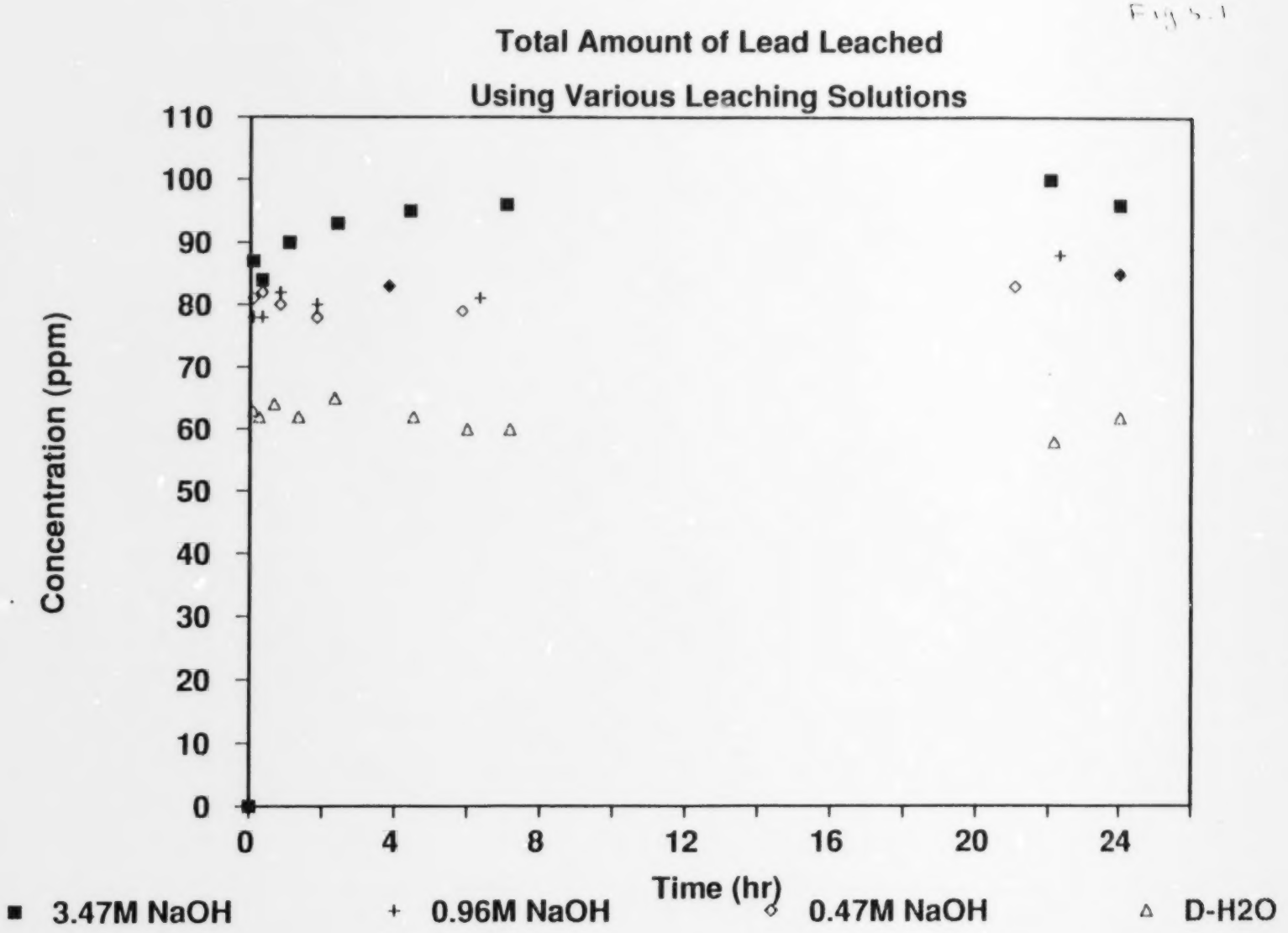
Table 5.3

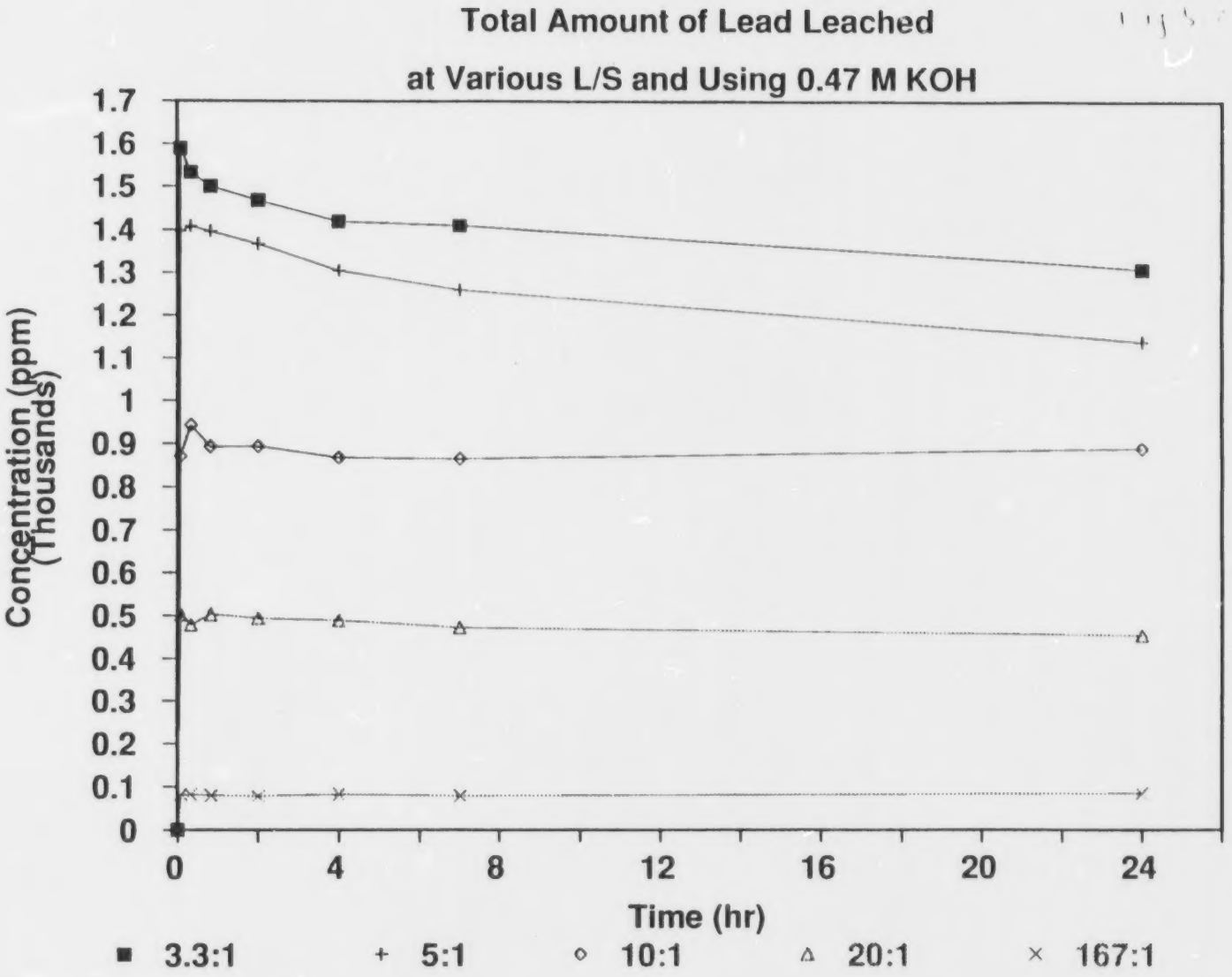
MOE Leachate Results

	pH		concentration (ppm)	
Time	sample A	sample B	sample A	sample B
15 min	11.80	11.84	38	40
24 hr	11.33	11.31	43	39
			45	39
		average:	42	39

Figures 5.1 to 5.6 are the key summary plots from the data.

Fig 5.1


Total Amount of Lead Leached
Using Various Leaching Solutions
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
Time (hr)
3.47M NaOH
0.96M NaOH
0.47M NaOH
D-H2O


Total Amount of Lead Leached
at Various L/S and Using 0.47 M KOH
Concentration (ppm)
(Thousands)
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
4
8
12
16
20
24
Time (hr)
3.3:1
5:1
10:1
20:1
167:1

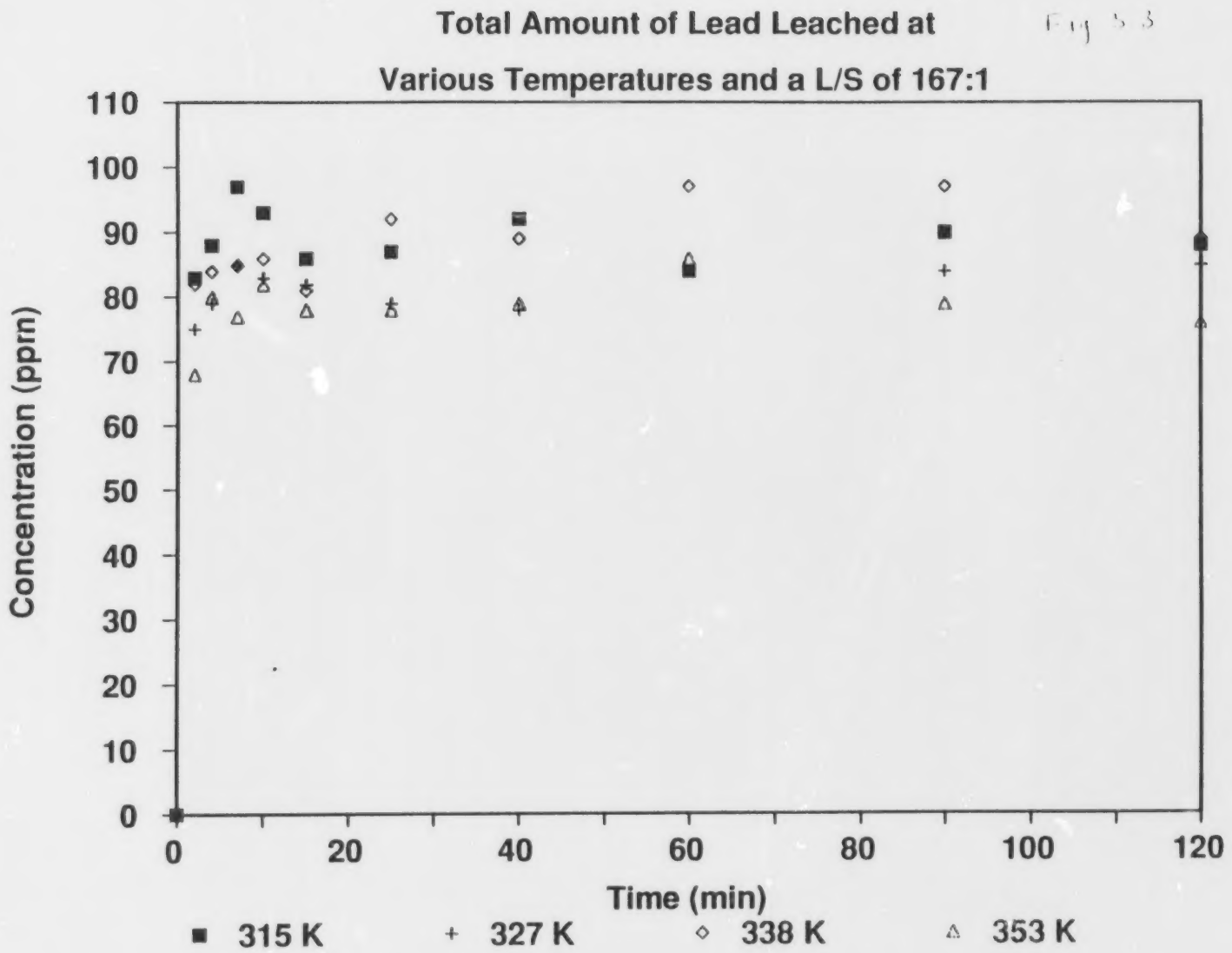
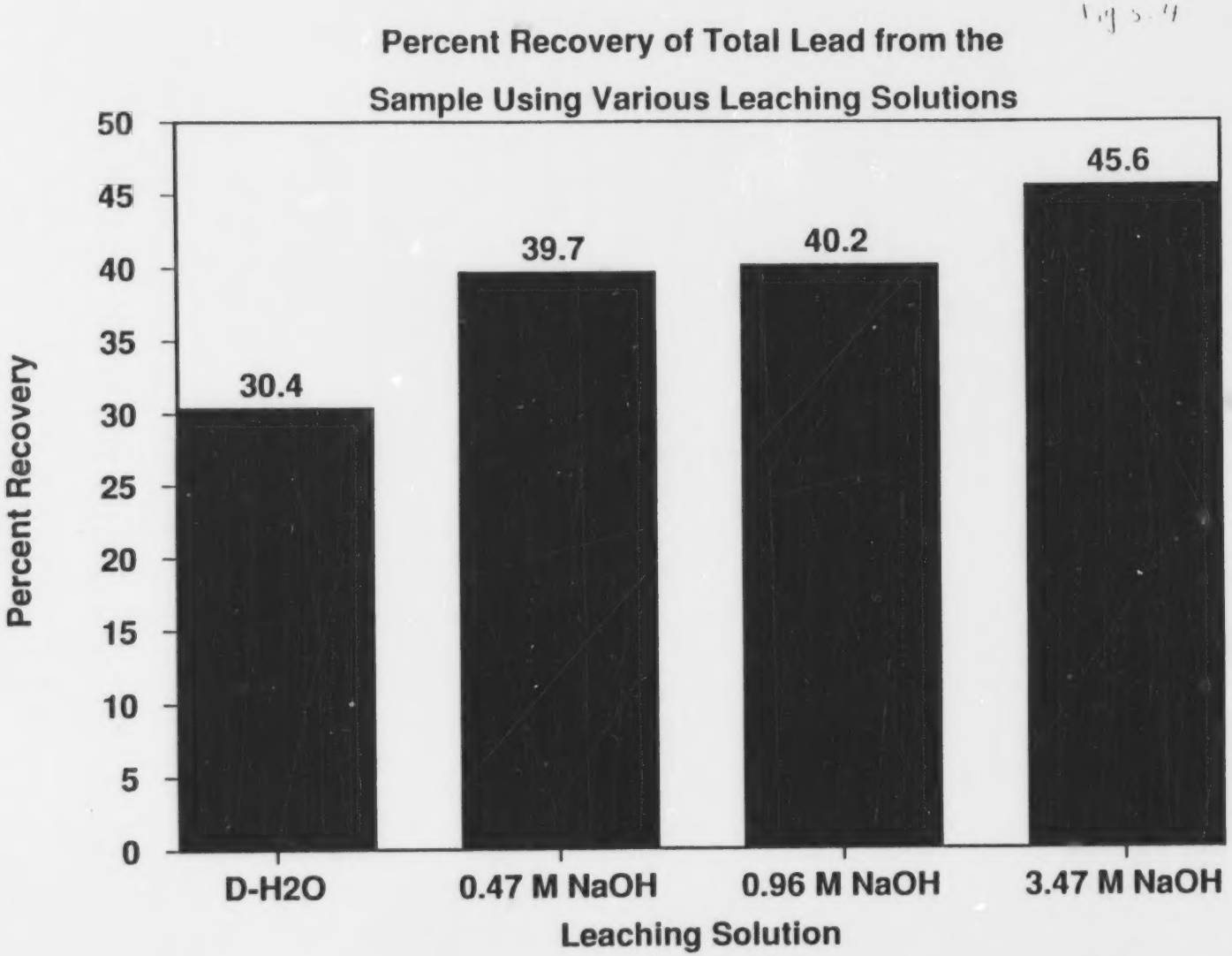
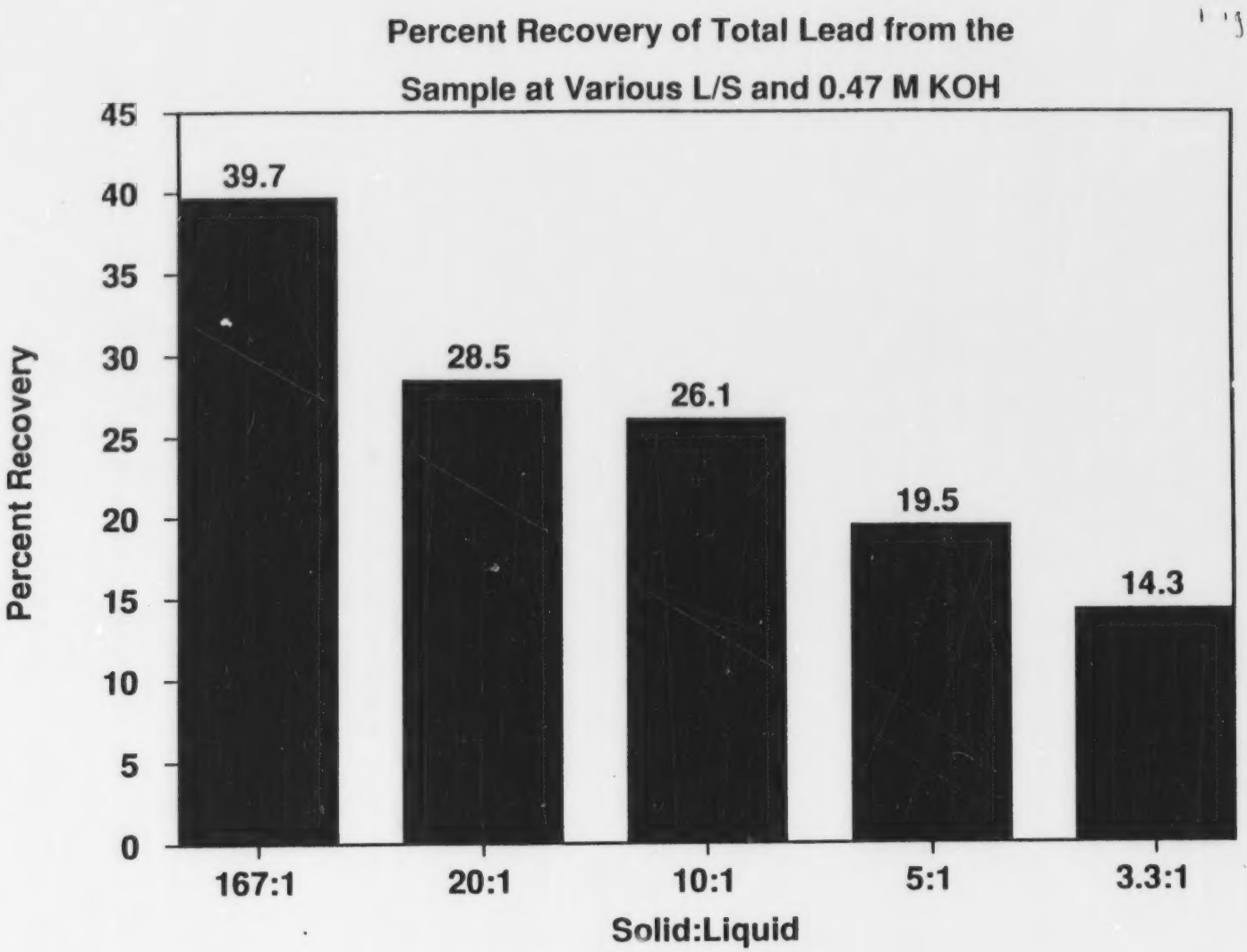
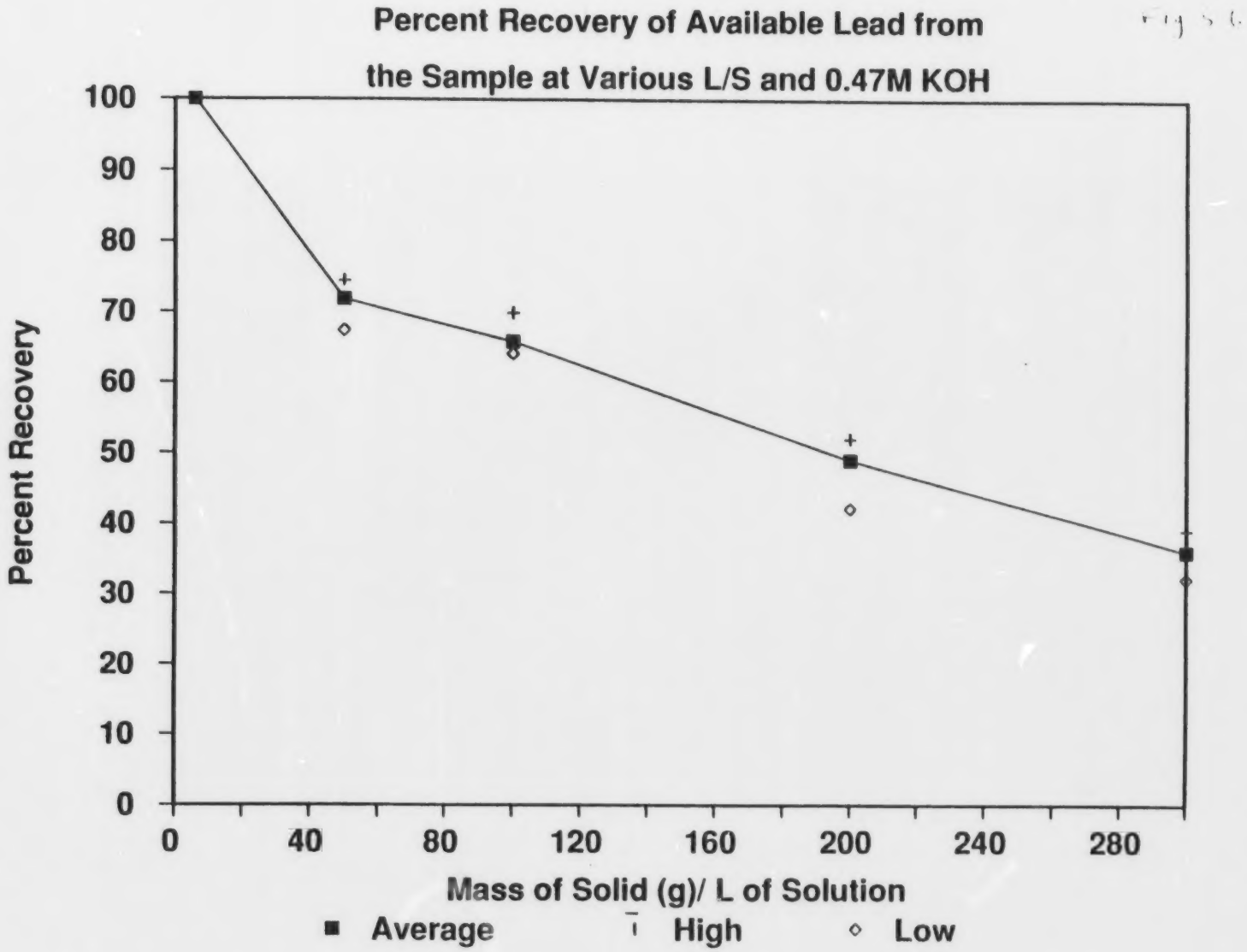

Total Amount of Lead Leached at
Various Temperatures and a L/S of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Time (min)
315 K
327 K
338 K
353 K

Fig 5.3


Percent Recovery of Total Lead from the
Sample Using Various Leaching Solutions
Percent Recovery
50
45
40
35
30
25
20
15
10
5
0
30.4
39.7
40.2
45.6
D-H2O
0.47 M NaOH
0.96 M NaOH
3.47 M NaOH
Leaching Solution


Percent Recovery of Total Lead from the
Sample at Various L/S and 0.47 M KOH
Percent Recovery
45
40
35
30
25
20
15
10
5
0
39.7
28.5
26.1
19.5
14.3
167:1
20:1
10:1
5:1
3.3:1
Solid:Liquid


Percent Recovery of Available Lead from
the Sample at Various L/S and 0.47M KOH
Percent Recovery
100
90
80
70
60
50
40
30
20
10
0
0
40
80
120
160
200
240
280
Mass of Solid (g)/ L of Solution
Average
High
Low

6.0 DISCUSSION

The experimental work consisted of three main experiments and some auxiliary work. For all the experiments, titrations were conducted at the conclusion of the 24 hour period in order to determine the effective hydroxide concentration of the waste which was indicative of the pH.

Initially, a glass electrode was used to measure the pH but the results appeared to be rather low so titrations were then conducted. The glass electrode works well only between pH values of 1 to 9. The pH range that was being used was from 12 to 14.5. Beyond a pH of 9, a phenomenon known as the alkaline error exists [24].

An electrode behaves as a site for proton transfer. As protons are acquired or lost, the potential at the surface of the electrode changes and a pH value is given by the meter. When a solution contains sodium ions or other cations, the electrode responds to these ions causing low pH readings. As the concentration of the cation increases, the error increases. Also, alkaline solutions destroy the surface of the glass electrode.

6.1 Alkalinity of the Leaching Solution

The first set of experiments dealt with varying the leaching solution. The temperature and the L/S ratio were held constant at 298 K and 167:1 respectively, for each experiment.

An obvious trend can be seen by examining Figure 5.1. As the alkalinity of the leaching solution increased, the amount of lead leached from the steel flue dust also increased in the 24 hour period. Approximately 62 ppm of lead was leached with the deionized water while 93 ppm was leached with the 3.47M NaOH. As illustrated by Figure 5.4 (also, refer to Appendix II) the percent recovery of the total lead in the sample increased from 30% to 46% as the alkalinity increased.

The pH corresponding to the deionized water and the 3.47M NaOH was about 12.1 and 14.5 respectively. This large range in pH may account for the almost 16% increase in the amount of lead leached from the steel flue dust when comparing the two leaching solutions. Figure 6.1 is a Pourbaix plot relating the total theoretical solubility of lead in the alkaline region. As illustrated in the plot, as the pH increased, the solubility also increased. Thus, in theory when the 3.47M NaOH solution was used as the leaching medium, the highest concentration should be observed which is what the results indicate.

6.2 Liquid to Solid Ratio

The second set of experiments examined the effects of five L/S ratios ranging from 3.3:1 to 167:1. The leaching solution chosen for these experiments was 0.47M KOH and the temperature was constant at 298 K. The results are shown in Figure 5.2.

As the L/S ratio decreased, ie., the mass of solid in 1L of solution increased, the concentration of lead leached from the sample increased. The range of concentrations was from 81 ppm for the L/S of 167:1 to 1462 ppm for the L/S of 3.3:1. The pH values did not change when the L/S ratio was changed. The pH was

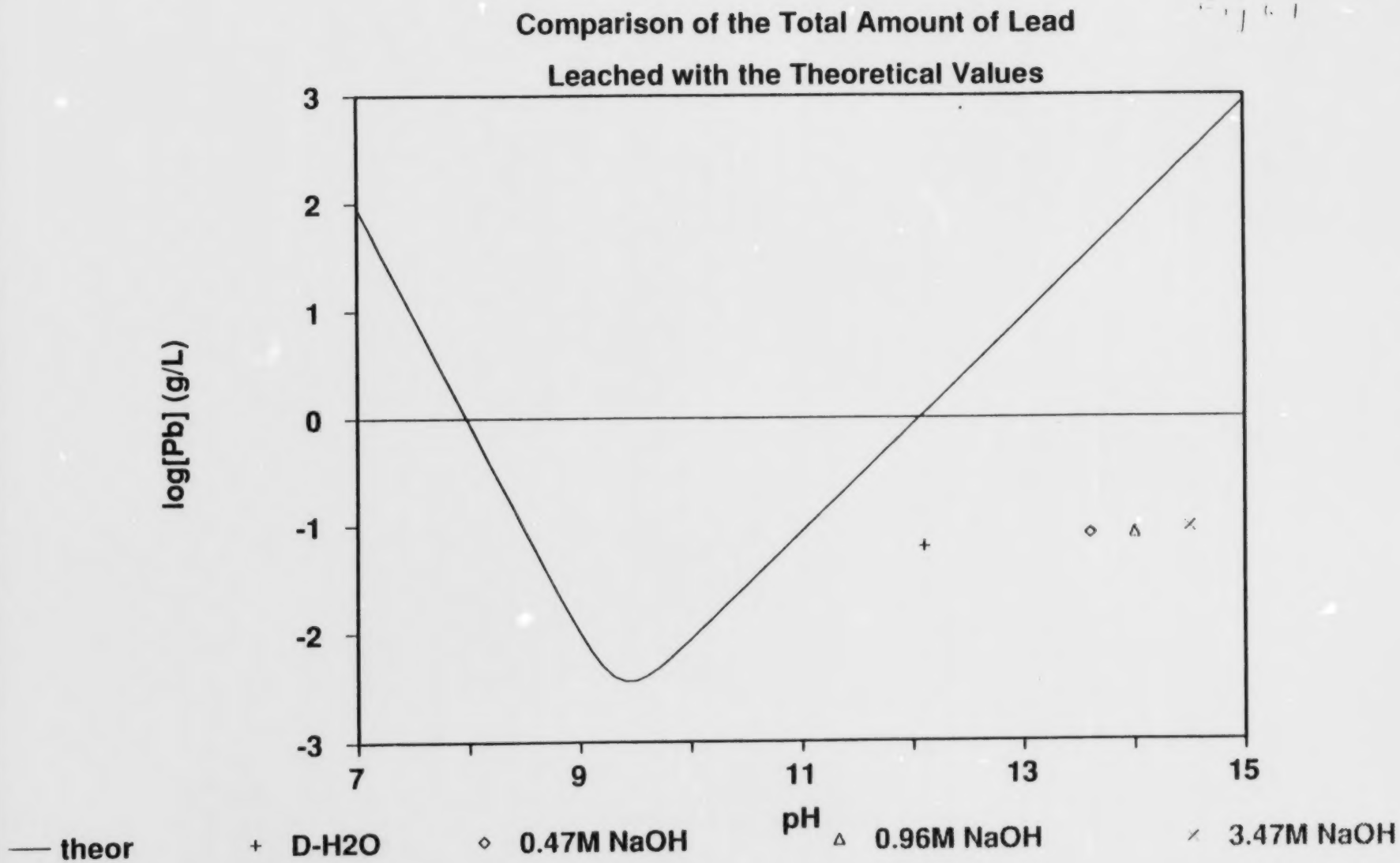
about 13.6 for each experiment. This observation was supported by Cote and Constable [15]. They found that the L/S ratio had no effect on the pH.

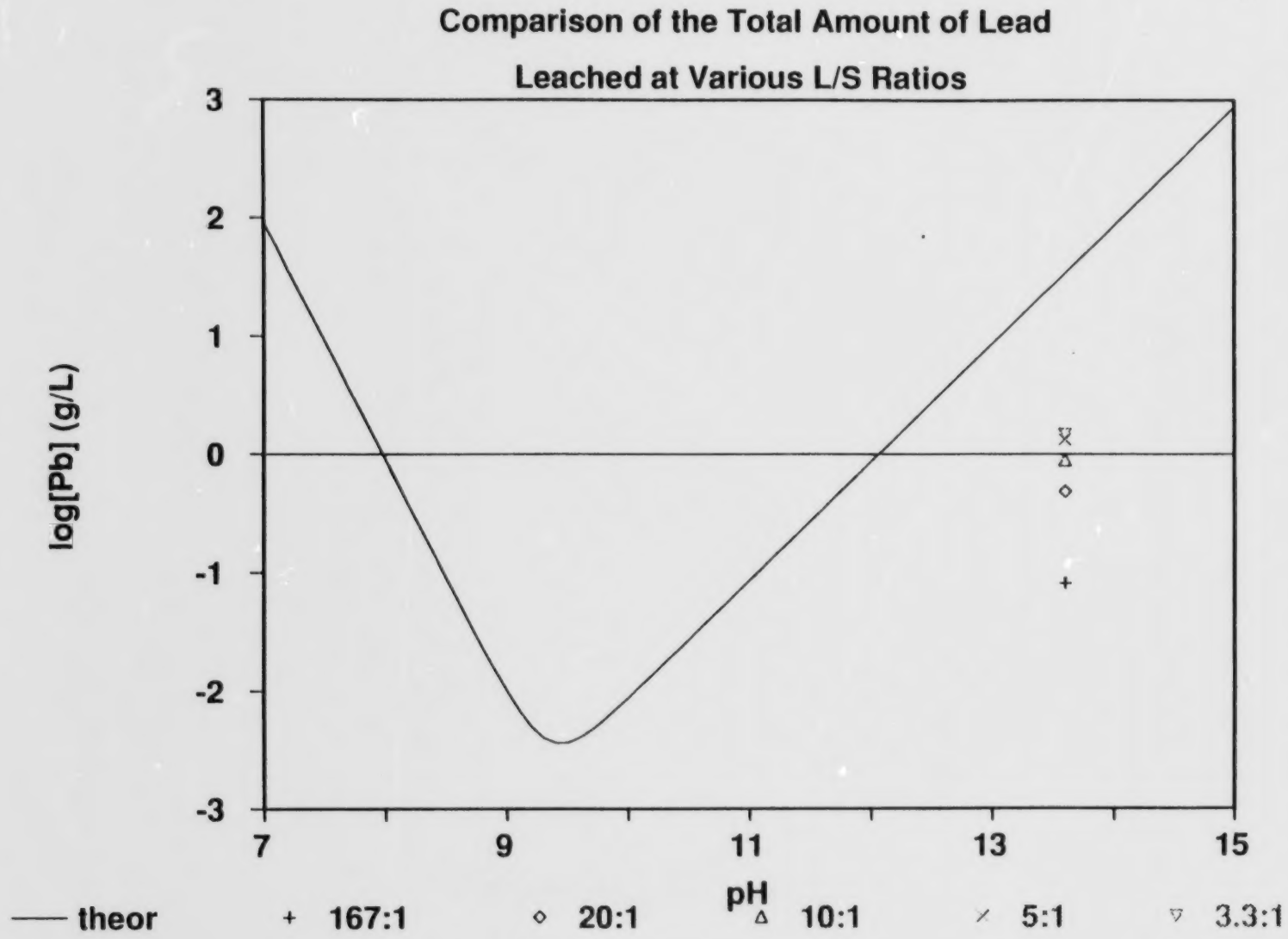
As Figure 5.5 illustrates, these values can be misleading. Although the 3.3:1 ratio yielded 1462 ppm, this value only represented 14.3% of the total lead in the steel flue dust. On the other hand, the high ratio of 167:1 resulted in a 39.7% recovery.

Not all of the lead was recovered because some of the lead was bound in stable ferrites and silicates [12]. Since the amount of available lead that could be leached out of the steel flue dust is important, these values were converted into a percent relating the availability of the lead. The results are shown in Figure 5.6.

The data seem to indicate that a linear relationship exists. Although the initial points are not linear, this may be due to experimental error. More data is needed in the lower L/S ratio region in order to conclude that this relationship is linear.

The trend observed in Figure 5.2 may be due to a couple of factors. The elevated concentrations may have been caused by the physical addition of more sample. As the L/S ratio decreased from 20:1 to 10:1 to 5:1, which corresponds to doubling the mass of the sample, the concentration did not double each time. Instead, the concentration went from 485 ppm to 889 ppm to 1325 ppm. Since this is not linear, another factor appears to be the controlling parameter. The average concentration for each ratio was also compared to the Pourbaix plot in Figure 6.2. Initially, their was a large increase in concentration but the increase diminished quickly as the L/S decreased. This seems to indicate that a solubility limit may exist in which case no matter how much sample is added, additional lead will not leach out of the steel flue dust.


Comparison of the Total Amount of Lead
Leached with the Theoretical Values
log[Pb] (g/L)
3
2
1
0
-1
-2
-3
7
9
11
13
15
pH
theor
D-H2O
0.47M NaOH
0.96M NaOH
3.47M NaOH


Comparison of the Total Amount of Lead
Leached at Various L/S Ratios
log[Pb] (g/L)
3
2
1
0
-1
-2
-3
7
9
11
13
15
pH
theor
167:1
20:1
10:1
5:1
3.3:1

6.3 Temperature

The final set of experiments examined the effects of temperature. The range investigated was from 298 K to 353 K. In each experiment, the L/S ratio was 167:1 and the leaching solution was 0.47M KOH. The results are plotted in Figure 5.3.

Most of the data lie in the same general range and no obvious pattern or trend can be observed from this data. Although the concentrations varied from a low of 67 ppm to a high of 102 ppm, this 35 ppm range was for approximately 80 samples. Also, the range for the average values was only 78 ppm to 89 ppm. An 11 ppm error can easily be associated with dilution and experimental errors.

The form of the Pourbaix equation for the solubility of lead is given by $\log[Pb^{++}] = \log(1/K) - 2pH$ (refer to Appendix III) where K is the equilibrium constant. To examine the effect that temperature has, recall that $K = \exp[nfE/RT]$. Since all the values in this equation remain the same except for temperature, K is proportional to $\exp(1/T)$. Given the range of temperatures that were examined, the factor $\exp(1/T)$ can be considered constant. Thus, temperature has negligible effects on the amount of lead leached from the steel flue dust as illustrated by Figure 5.3.

6.4 MOE Leach Test

The MOE leach test was conducted as specified in the official procedure [3]. A treated sample from the 5:1 experiment was used. As the results in Table 5.3 illustrate, the average concentration was about 41 ppm or 1.5% of the available lead. Originally, 1325 ppm leached from an untreated sample or 49% of the available lead.

If the assumption is made that 1.5% is a common residual amount for all the L/S ratios, then the 20:1 ratio sample would yield a concentration of about 10 ppm and the 167:1 ratio sample would yield a concentration of only 1 ppm. Interpolating between these ratios indicates that any ratio greater than 100:1 would pass the MOE leach test. Further tests are needed to confirm this assumption.

6.5 Miscellaneous

Some minor experiments were also conducted to substantiate some assumptions that were made during the experiments. The effects of mixing were checked by conducting three runs using magnetic stirrers instead of the tumbling apparatus. As indicated by the results (Table 13 in Appendix IV), the type of mixing had no appreciable effects. The results were within 7 ppm.

The duration of the experiment was also investigated. A 167:1 ratio sample was placed in 1L of 0.96M KOH and allowed to stay in contact with the leaching solution for 25 days. At the end of the period, the average concentration was about 100 ppm. A concentration of 92 ppm after 5 days and 82 ppm after 1 day were obtained under similar conditions. Although the concentration did increase by about 20 ppm, running experiments for more than 24 hours in duration would not be feasible.

In general, most of the data followed a pattern. The data that deviated from the trends was due to experimental errors which could not be controlled. Since all the samples were diluted by either a factor of 5, 10, or 15, an error of 2.5 ppm to 7.5 ppm was unavoidable just from the accuracy of the instrument. The AAS had

an accuracy of 0.5 ppm. Also, the errors occurred when diluting the samples.

7.0 CONCLUSIONS

The following conclusions are drawn:

1. A very alkaline leaching solution yields the highest concentration of lead leached from the steel flue dust.

2. This corresponds to 45.6% by weight of the total lead in the sample.

3. The concentration of lead leached from the steel flue dust increases as the L/S ratio decreases.

4. The percent recovery of the available lead increases as the L/S ratio increases.

5. Temperature has a negligible effect on the amount of lead leached at the concentrations studied.

6. L/S ratios of 100:1 or greater may pass the leach test.

8.0 RECOMMENDATIONS

1. Further work should be conducted to study the effects of multiple sequential leaching.

2. A counter current continuous process should be designed and investigated in order to compare with a batch process.

3. The elements zinc and cadmium should be included with lead in the leaching process extraction.

4. Further work should concentrate on meeting the MOE leachate requirements.
5. Experiments should be performed to check the theory that L/S ratios of 100:1 or greater would pass the MOE leachate requirements.

9.0 REFERENCES

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Experimental Data

APPENDIX I: AAS Calibration Curve

Model: Perkin - Elmer 703 AAS
Sensitivity: 0.5 ppm
Wavelength: about 284 nm
Slit: 4

Absorbance	ppm
0.002	0
0.077	10
0.161	20
0.319	40

Regression Output

Constant	0
Std Err of Y Est	0.296840
R Squared	0.999697
No. of Observations	4
Degrees of Freedom	3
X Coefficient(s)	125.3947
Std Err of Coef.	0.812308

Equation of the line was forced through the origin accounting for the value of 0 for the constant.

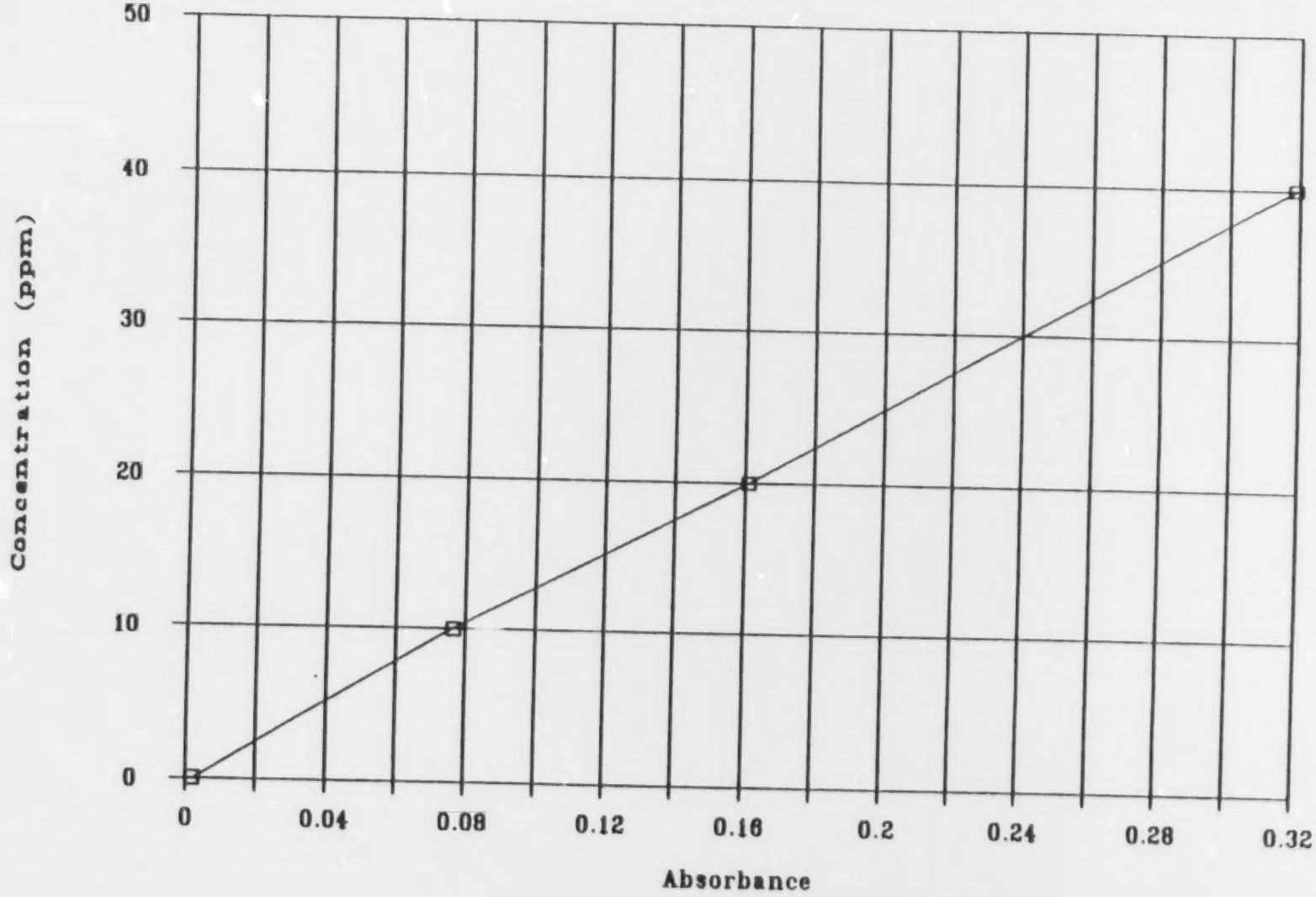
Equation of the line is: $Y = mX + b$

where: Y = concentration in ppm
m = slope of line
X = absorbance
b = y intercept

Therefore, the equation of the line in Figure I is:

$Y = 125.3974*X$

Figure 1: AAS CALIBRATION CURVE



APPENDIX II: Percent Recovery of Total and Available Lead in the Steel Flue Dust

1. Different Leaching Medias

- assume that the sample is 3.4% lead by weight [6]
- each experiment had 6g of sample in 1L of solution

max concentration = (6g)*(34mg Pb/g sample)*(1/1L) = 204 ppm

Therefore, percent recovery of total Pb for the 3.47M experiment is:

(93ppm/204ppm)*100% = 45.6%

2. Different L/S Ratios

- percent recovery was calculated as above
- the highest recovery was 39.7% for the 167:1 experiment
- since this was the highest, it was chosen as the reference to calculate the percent of the available Pb that leached

Therefore, percent recovery of the available Pb for the 20:1 exp't is:

[(485ppm)/(50g*34mg Pb/g)]*100%/39.7% = 71.9%

APPENDIX III: Solubility of Lead Using Pourbaix Data

Equations used from Pourbaix [23]:

1. $Pb^{++} + H_2O = PbO + 2H^+$ $\quad \log[Pb^{++}] = 13.64 - 2pH$

$$[Pb^{++}] = 10^{(13.64-2pH)} \quad (1)$$

2. $PbO + H_2O = HPbO_2^- + H^+$ $\quad \log[HPbO_2^-] = -14.38 + pH$

$$[HPbO_2^-] = 10^{(-14.38+pH)} \quad (2)$$

3. $Pb^{+4} + 2H_2O = PbO_2 + 4H^+$ $\quad \log[Pb^{+4}] = -8.26 - 4pH$

$$[Pb^{+4}] = 10^{(-8.26-4pH)} \quad (3)$$

4. $PbO_2 + H_2O = PbO_3^{--} + 2H^+$ $\quad \log[PbO_3^{--}] = -31.32 + 2pH$

$$[PbO_3^{--}] = 10^{(-31.32+2pH)} \quad (4)$$

Recall: $nfE = \Delta G = RT\ln K$

From (1): $K = [H^+]2/[Pb^{++}]$

$[Pb^{++}] = [H^+]2/K$

$\log[Pb^{++}] = \log(1/K) + 2\log(H^+)$

$= \log(1/K) - 2pH$

Total [Pb] = sum of (1) to (4)

$[Pb] = 10^{(13.64-2pH)} + 10^{(-14.38+pH)} + 10^{(-8.26-4pH)} + 10^{(-31.32+2pH)}$

= mol/L * 207.2 g/mol

= g/L

Table III.1

Total Lead Solubility in the Alkaline Range

pH	[Pb]	log[Pb]
7	90.4461	1.956
7.2	36.0072	1.556
7.4	14.3348	1.156
7.6	5.7068	0.756
7.8	2.2720	0.356
8	0.9045	-0.044
8.2	0.3602	-0.443
8.4	0.1436	-0.843
8.6	0.0574	-1.241
8.8	0.0233	-1.633
9	0.0099	-2.004
9.2	0.0050	-2.304
9.4	0.0036	-2.443
9.6	0.0040	-2.397
9.8	0.0057	-2.246
10	0.0087	-2.059
10.2	0.0137	-1.862
10.4	0.0217	-1.663
10.6	0.0344	-1.464
10.8	0.0545	-1.264
11	0.0864	-1.064
11.2	0.1369	-0.864
11.4	0.2170	-0.664
11.6	0.3439	-0.464
11.8	0.5450	-0.264
12	0.8638	-0.064
12.2	1.3690	0.136
12.4	2.1697	0.336
12.6	3.4388	0.536
12.8	5.4503	0.736
13	8.6385	0.936
13.2	13.6921	1.136
13.4	21.7028	1.337
13.6	34.4024	1.537
13.8	54.5386	1.737
14	86.4745	1.937
14.2	137.1448	2.137
14.4	217.5908	2.338
14.6	345.4382	2.538
14.8	548.9396	2.740
15	873.6706	2.941

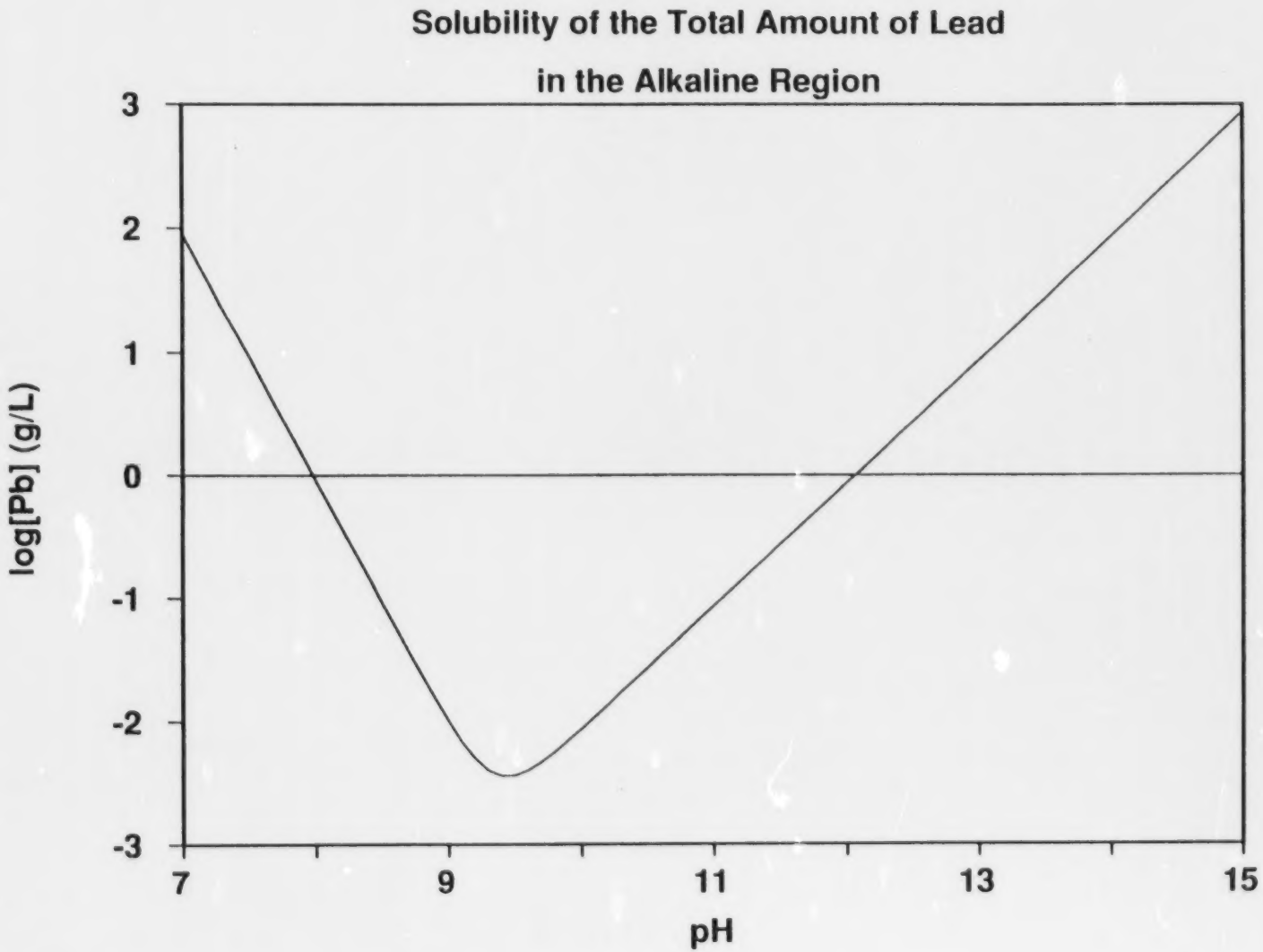

Solubility of the Total Amount of Lead
in the Alkaline Region
log[Pb] (g/L)
3
2
1
0
-1
-2
-3
7
9
11
13
15
pH

Table 1: Amount of Lead Leached Using Deionized Water and a L/S Ratio of 167:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	61	64	63	63
0.250	62	60	63	62
0.667	64	64	65	64
1.333	55	66	64	62
2.333	63	67	65	65
4.500	63	62	61	62
6.000	59	61	61	60
7.167	58	61	62	60
22.167	53	61	61	58
24	65	61	61	62
			average:	62
pH:	12.1	12.14	12.13	

Table 2: Amount of Lead Leached Using 0.472 M NaOH and a L/S Ratio of 167:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	79	81	83	81
0.333	82	82	82	82
0.833	74	81	84	80
1.833	73	78	84	78
3.833	82	77	90	83
5.833	79	74	83	79
21.083	80	78	91	83
24	-	-	85	85
168	-	-	96	96
			average:	81

Table 3: Amount of Lead Leached Using 0.96 M NaOH and a L/S Ratio of 167:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	83	77	75	78
0.333	84	82	69	78
0.833	80	84	81	82
1.833	73	81	85	80
3.833	85	85	80	83
6.333	84	73	87	81
22.333	88	93	83	88
24	88	82	85	85
120	95	93	87	92
			average:	82

Table 4: Amount of Lead Leached Using 3.47 M NaOH and a L/S Ratio of 167:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	93	84	84	87
0.333	90	77	86	84
1.083	87	90	92	90
2.411	91	94	95	93
4.417	101	92	93	95
7.083	101	91	97	96
22.083	101	98	102	100
24	102	89	97	96
			average:	93
pH:	14.54	14.53	14.53	

Table 5: Amount of Lead Leached Using 0.47 M KOH and a L/S Ratio of 20:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	484	513	513	503
0.333	438	493	507	479
0.833	480	526	504	503
2.000	503	531	451	495
4.000	473	517	479	490
7.000	432	505	482	473
24	425	479	460	455
			average:	485
pH:	13.65	13.65	13.65	

Table 6: Amount of Lead Leached Using 0.47 M KOH and a L/S Ratio of 10:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	784	924	901	870
0.333	839	1079	914	944
0.833	818	912	950	893
2.000	923	919	843	895
4.000	743	917	944	868
7.000	744	877	977	866
24	834	971	864	890
			average:	889
pH:	13.63	13.64	13.64	

Table 7: Amount of Lead Leached Using 0.47 M KOH and a L/S Ratio of 5:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	1341	1470	1376	1396
0.333	1336	1477	1412	1408
0.833	1323	1483	1381	1396
2.000	1326	1421	1358	1368
4.000	1222	1277	1420	1306
7.000	1246	1309	1226	1260
24	1064	1200	1152	1139
			average:	1325
pH:	13.59	13.61	13.60	

Table 8: Amount of Lead Leached Using 0.47 M KOH and a L/S Ratio of 3.3:1

Time (hr)	Concentration of Pb (ppm) sample A	sample B	sample C	Average
0	0	0	0	0
0.083	1695	1588	1483	1589
0.333	1562	1490	1551	1534
0.833	1545	1479	1479	1501
2.000	1364	1513	1531	1469
4.000	1423	1417	1421	1420
6.500	1368	1450	1412	1410
24	1190	1349	1389	1309
			average:	1462
pH:	13.56	13.58	13.54	

Table 9: Amount of Lead Leached at a Temperature of 42 C
Using 0.47 M KOH and a L/S Ratio of 167:1

Time (min)	Concentration (ppm) sample A	sample B	Average (ppm)	Temperature A	B
0	0	0	0	-	-
2	83	82	83	41.0	42.5
4	89	86	88	41.5	42.5
7	99	94	97	42.5	40.5
10	97	88	93	43.0	41.5
15	86	85	86	42.5	40.0
25	81	92	87	40.5	39.0
40	89	94	92	44.0	44.0
60	79	89	84	42.0	42.5
90	88	91	90	47.0	40.5
120	85	91	88	44.0	43.5
pH:	13.67	13.67	average:	42.8	41.7
			std dev:	1.9	1.6

Table 10: Amount of Lead Leached at a Temperature of 54 C
Using 0.47 M KOH and a L/S Ratio of 167:1

Time (min)	Concentration (ppm) sample A	sample B	Average (ppm)	Temperature A	B
0	0	0	0	-	-
2	78	71	75	50.0	56.0
4	85	73	79	52.5	56.0
7	87	82	85	54.5	55.0
10	84	82	83	56.0	54.0
15	80	84	82	58.0	53.0
25	79	79	79	56.5	51.0
40	75	80	78	54.0	50.0
60	86	82	84	50.0	52.5
90	86	81	84	58.0	56.0
120	84	85	85	52.0	50.0
pH:	13.64	13.65	average:	54.2	53.4
			std dev:	3.0	2.4

Table 11: Amount of Lead Leached at a Temperature of 65 C Using 0.47 M KOH and a L/S Ratio of 167:1

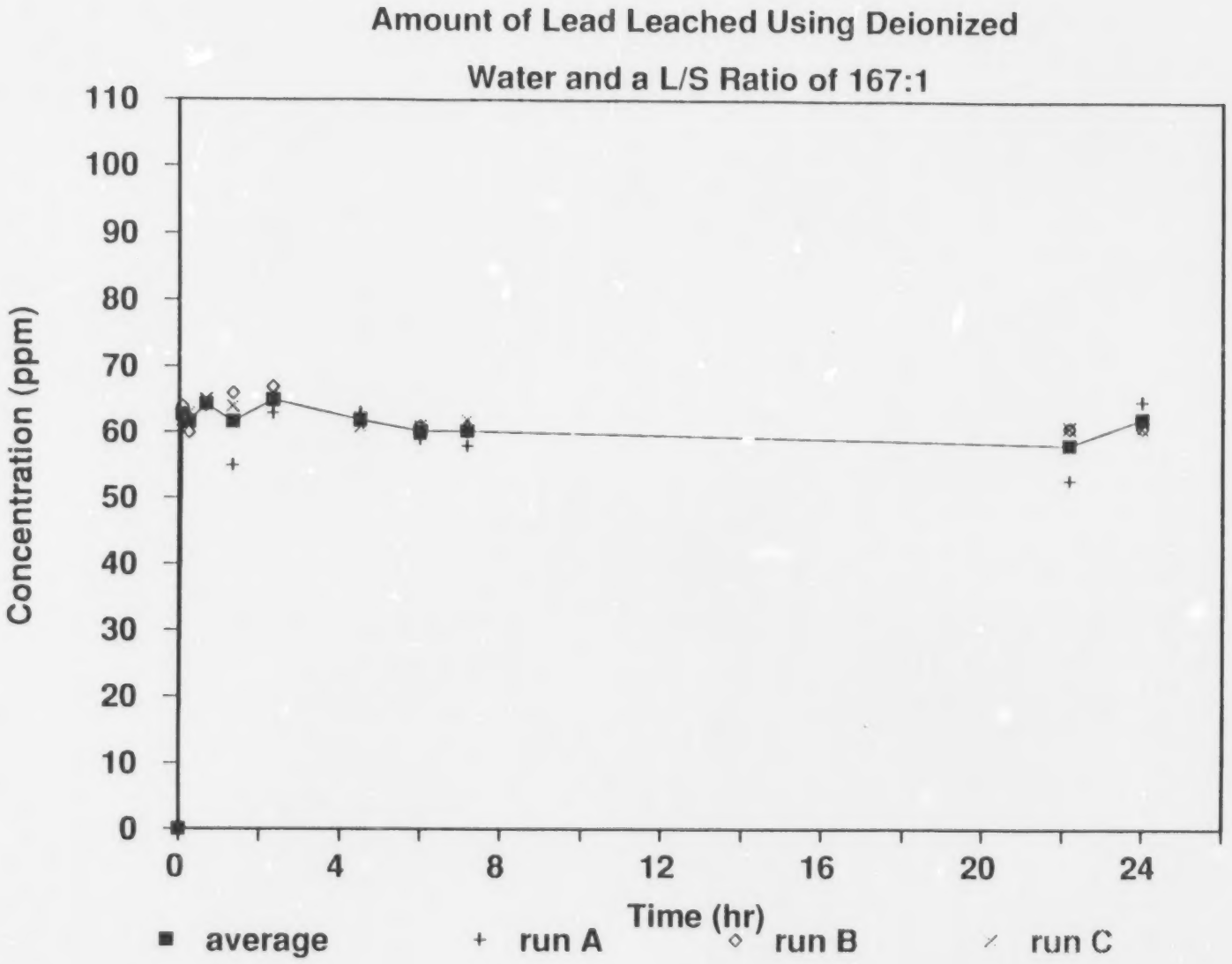
Time (min)	Concentration (ppm) sample A	sample B	Average (ppm)	Temperature A	B
0	0	0	0	-	-
2	80	84	82	65.5	64.5
4	82	86	84	64.5	65.0
7	85	84	85	63.5	65.5
10	85	87	86	63.0	65.0
15	79	82	81	63.0	63.5
25	94	89	92	64.0	60.0
40	90	87	89	65.5	65.0
60	92	102	97	67.0	71.0
90	94	101	98	60.0	62.0
120	92	86	89	67.5	64.0
pH:	13.67	13.65	average:	64.4	64.6
			std dev:	2.2	2.8

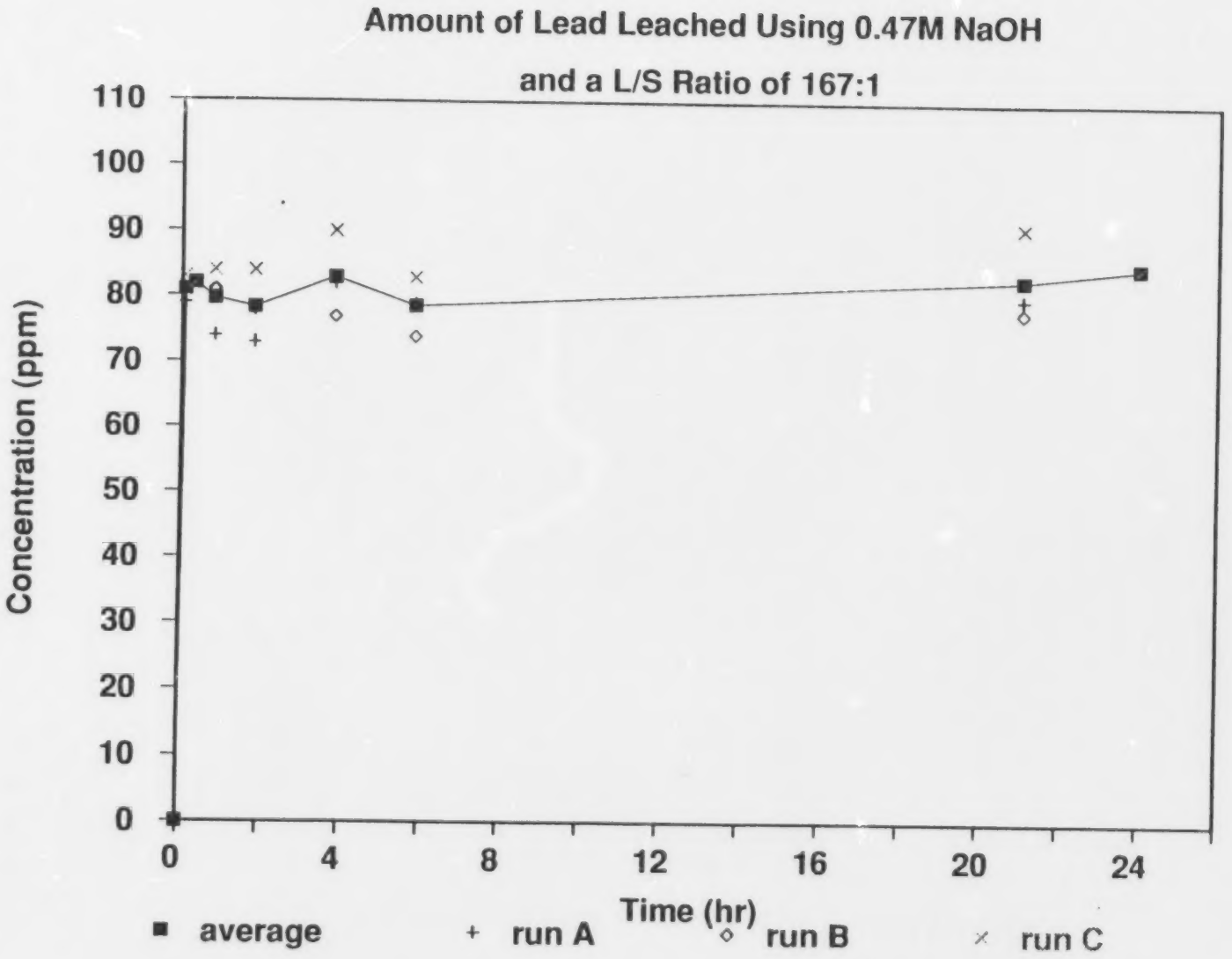
Table 12: Amount of Lead Leached at a Temperature of 80 C Using 0.47 M KOH and a L/S Ratio of 167:1

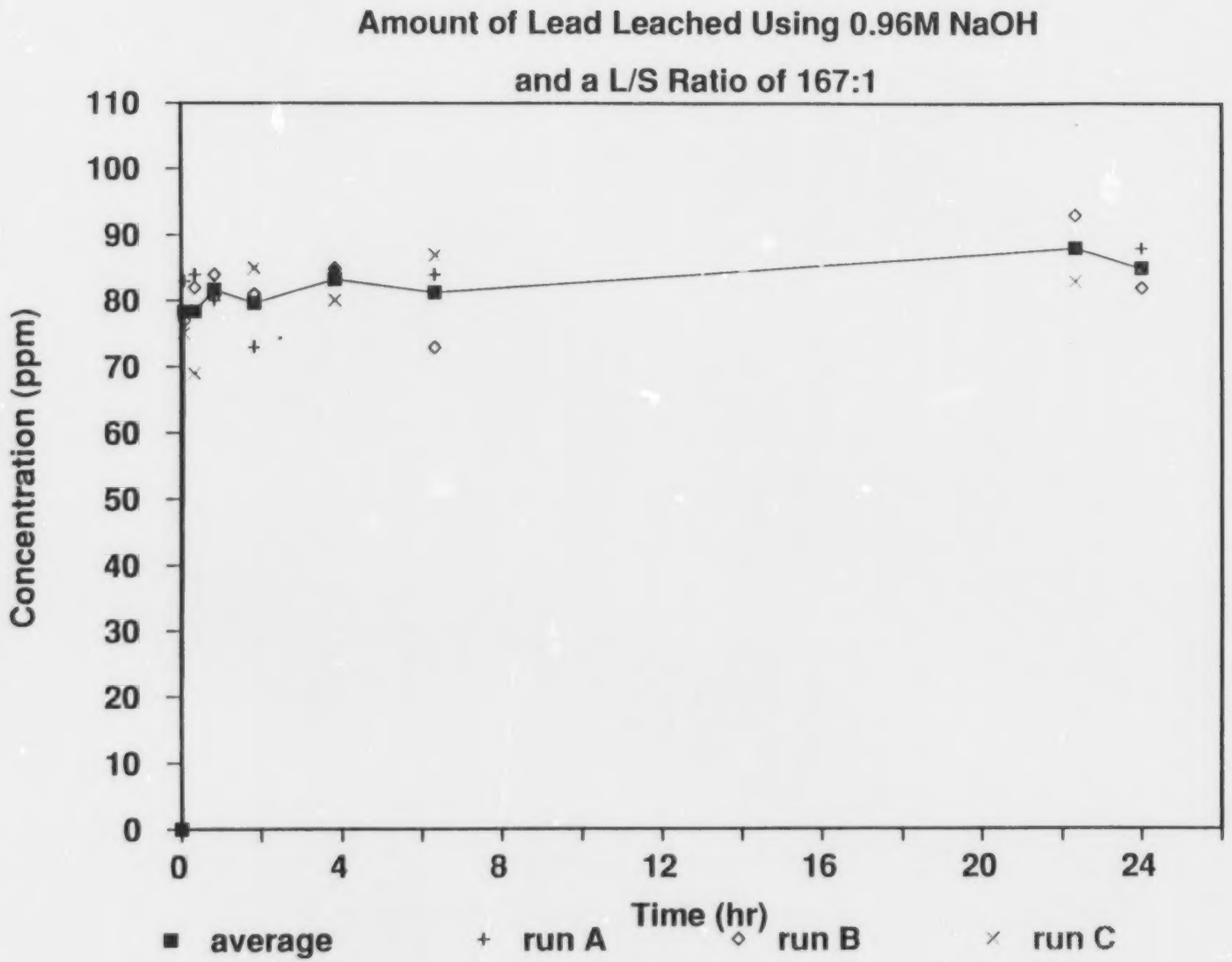
Time (min)	Concentration (ppm) sample A	sample B	Average (ppm)	Temperature A	B
0	0	0	0	-	-
2	68	67	68	83.5	79.0
4	76	84	80	83.0	78.5
7	76	78	77	80.0	78.5
10	85	79	82	79.5	79.0
15	74	82	78	81.0	80.5
25	78	78	78	87.0	80.0
40	81	77	79	80.0	76.0
60	92	80	86	77.0	81.0
90	78	80	79	78.0	76.0
120	87	65	76	81.0	79.0
pH:	13.68	13.67	average:	81.0	78.8
			std dev:	2.9	1.7

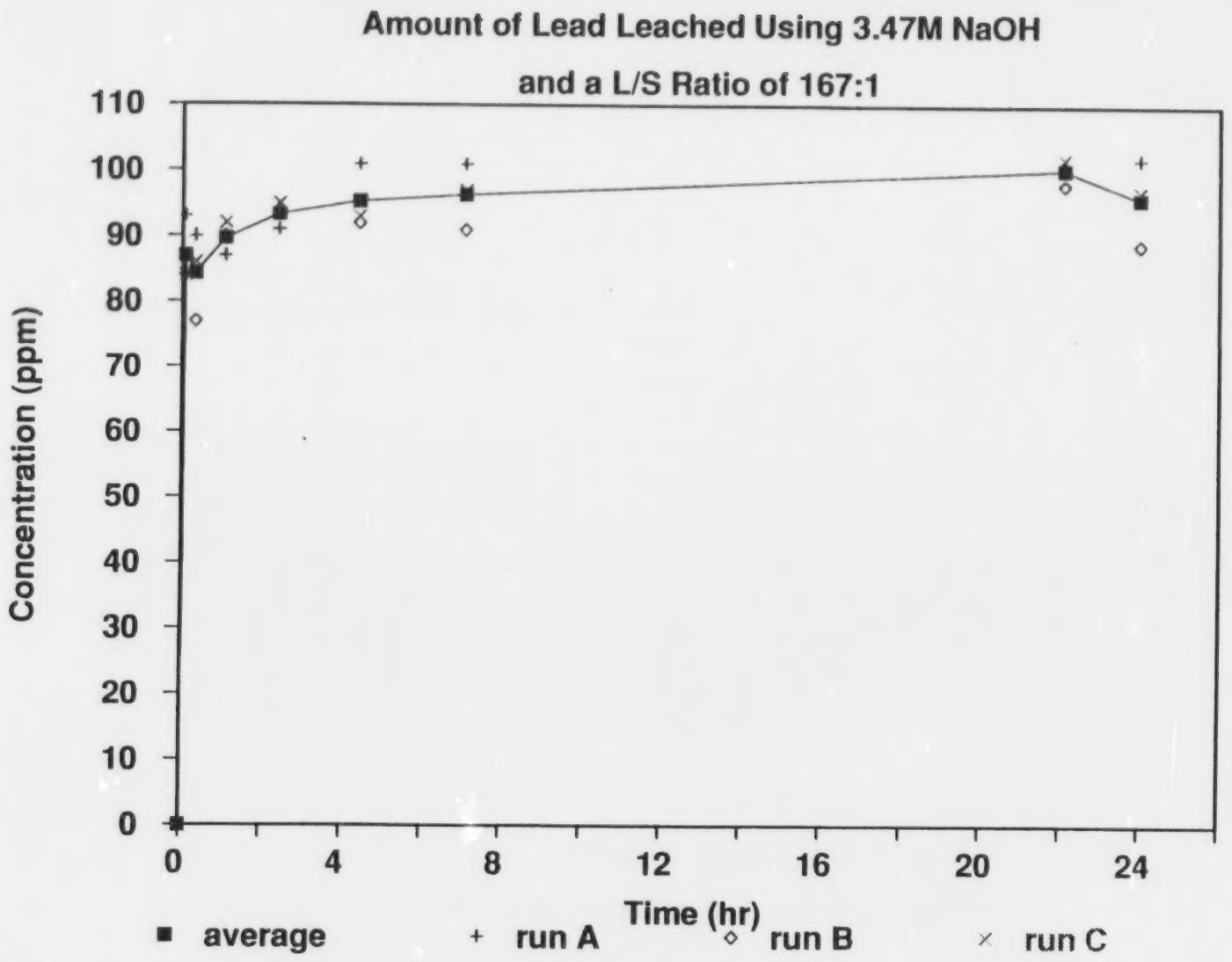
Table 13: Amount of Lead Leached Using Magnetic Stirrers and at a L/S Ratio of 167:1

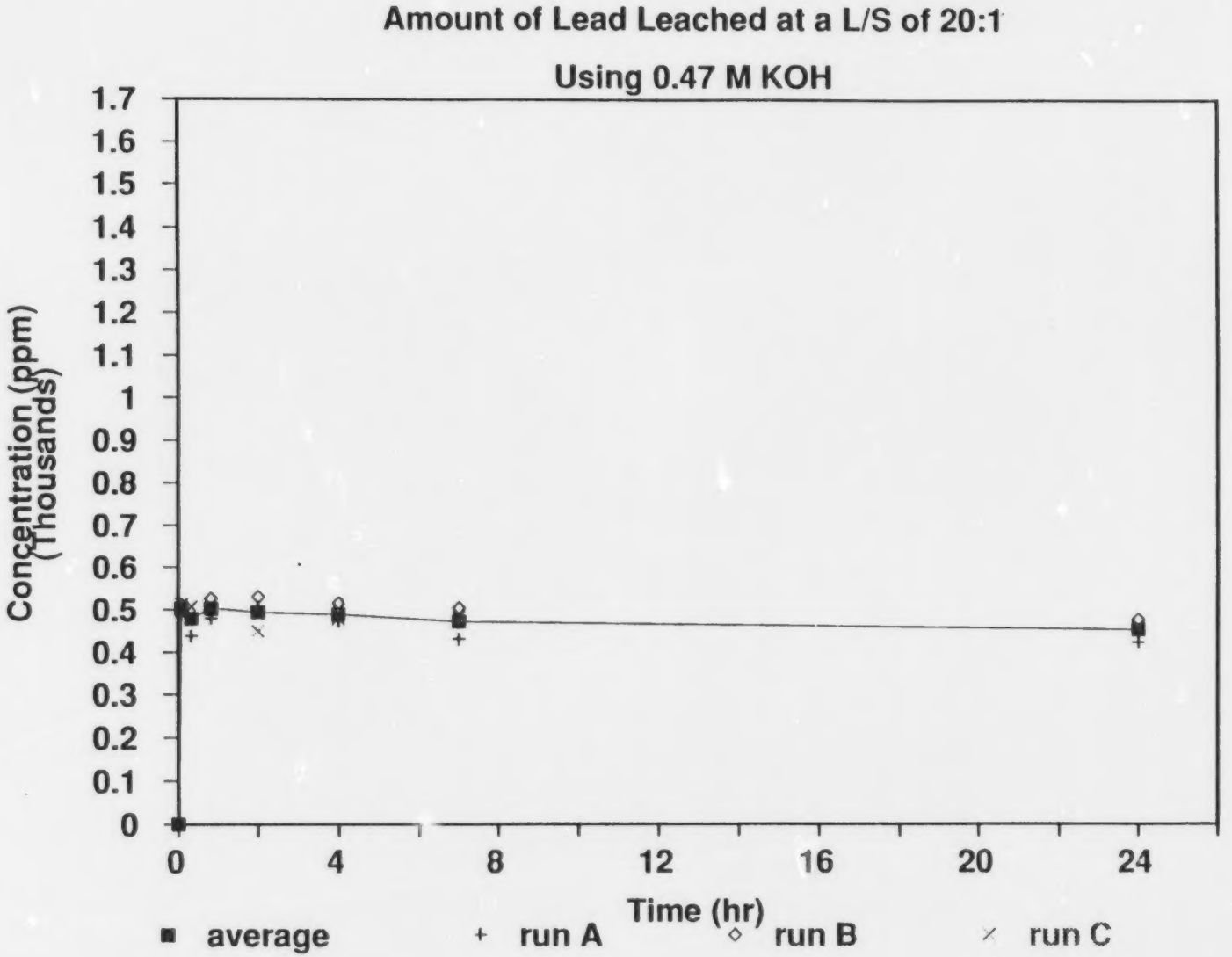
Time (hr)	Concentration of Pb (ppm) 3.47 M	0.96 M	0.47 M
0	0	0	0
0.083	73	74	75
0.333	82	77	66
0.833	83	77	78
1.833	91	80	77
3.833	86	73	-
6.833	90	80	79
24	99	91	89
average:	86	79	77
pH:	14.54	14.01	13.69

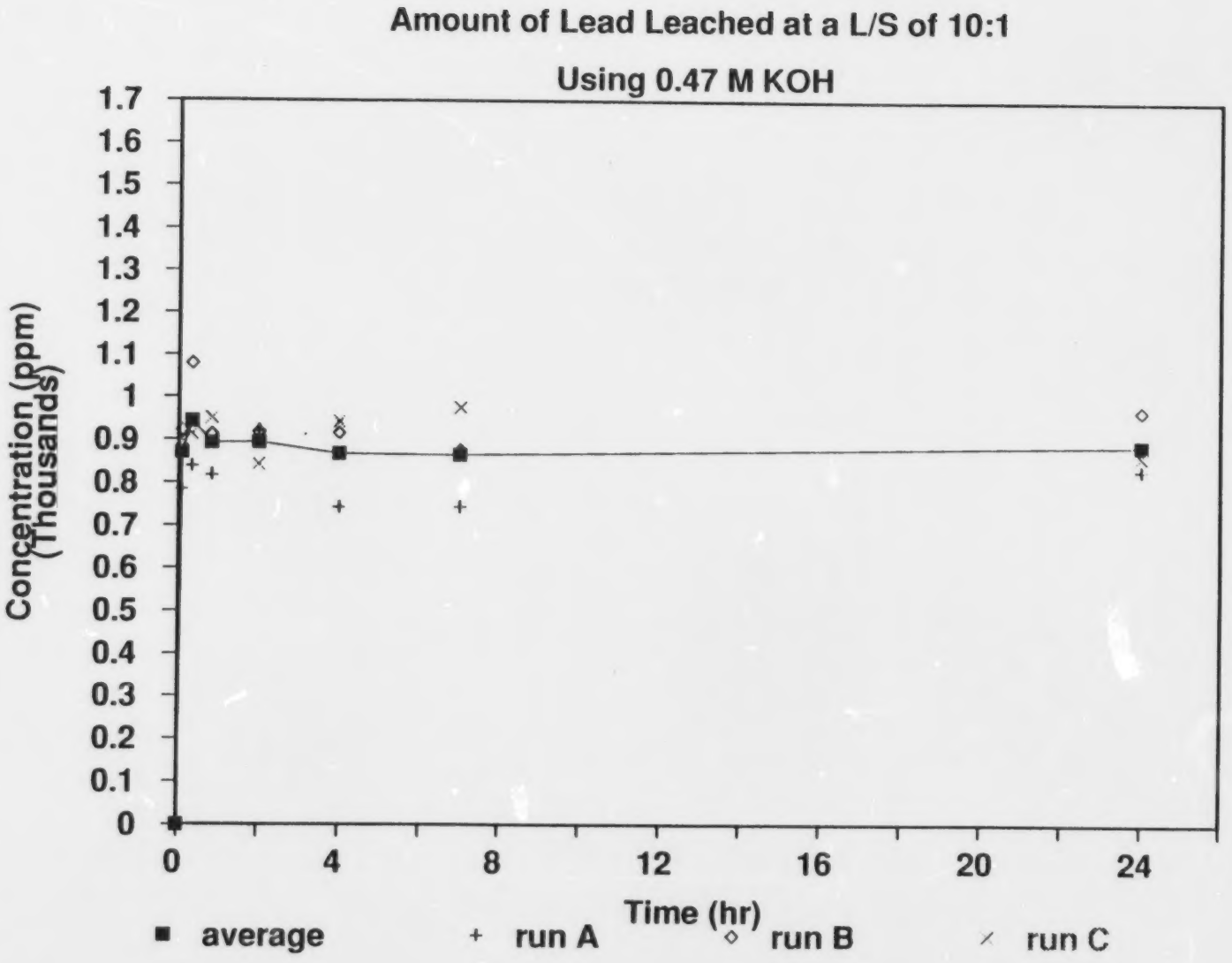

Amount of Lead Leached Using Deionized Water and a L/S Ratio of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

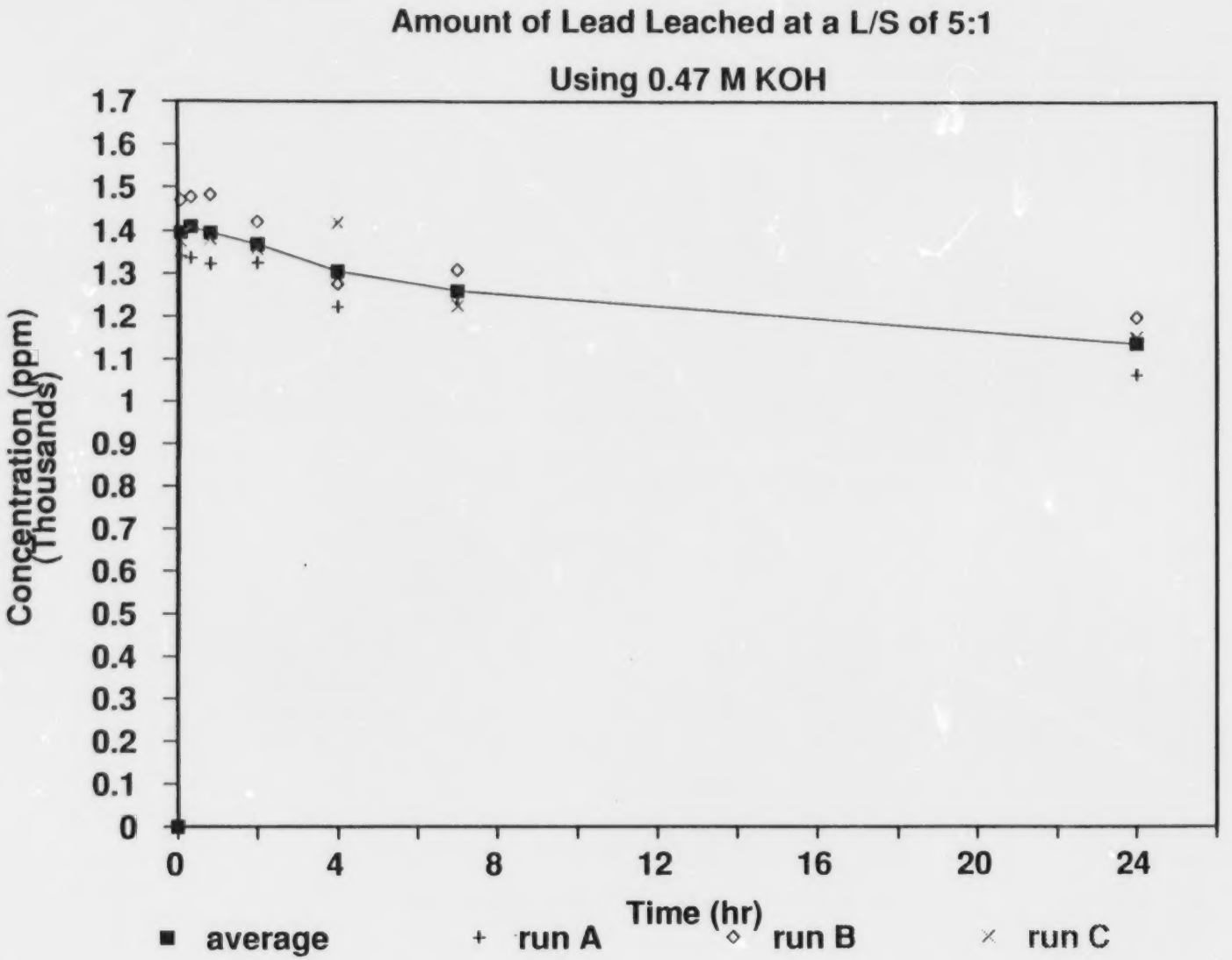

Amount of Lead Leached Using 0.47M NaOH
and a L/S Ratio of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

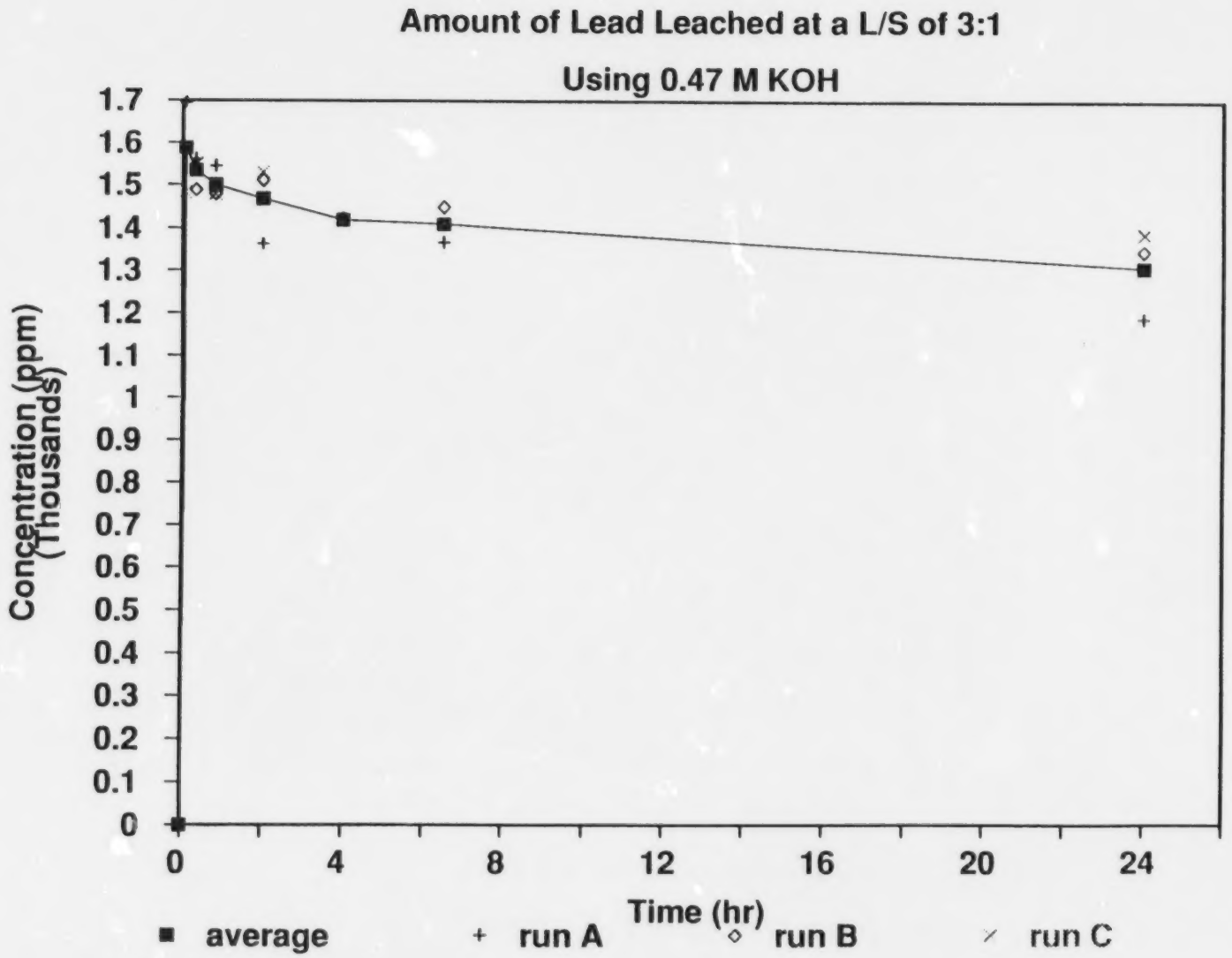

Amount of Lead Leached Using 0.96M NaOH
and a L/S Ratio of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

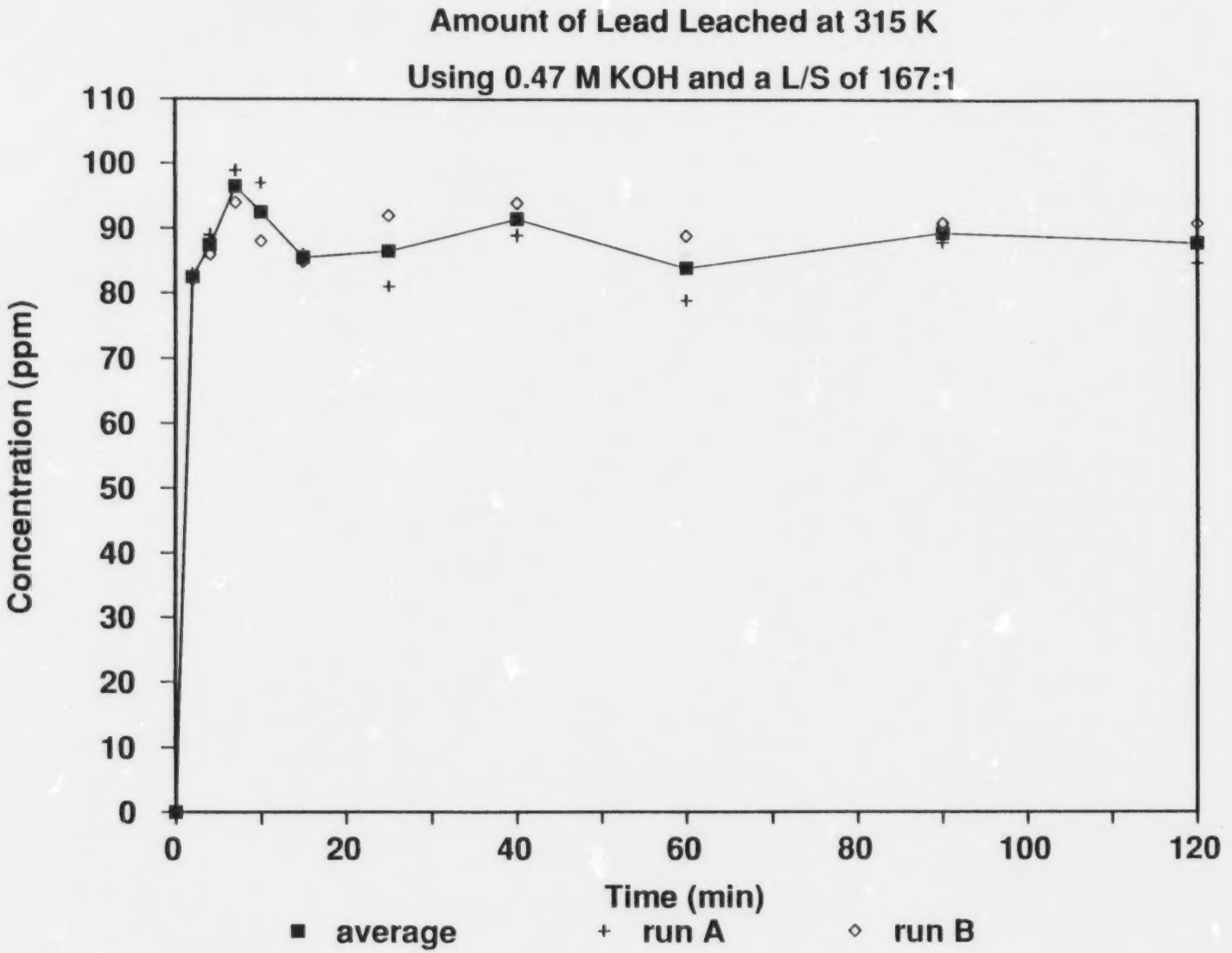

Amount of Lead Leached Using 3.47M NaOH
and a L/S Ratio of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

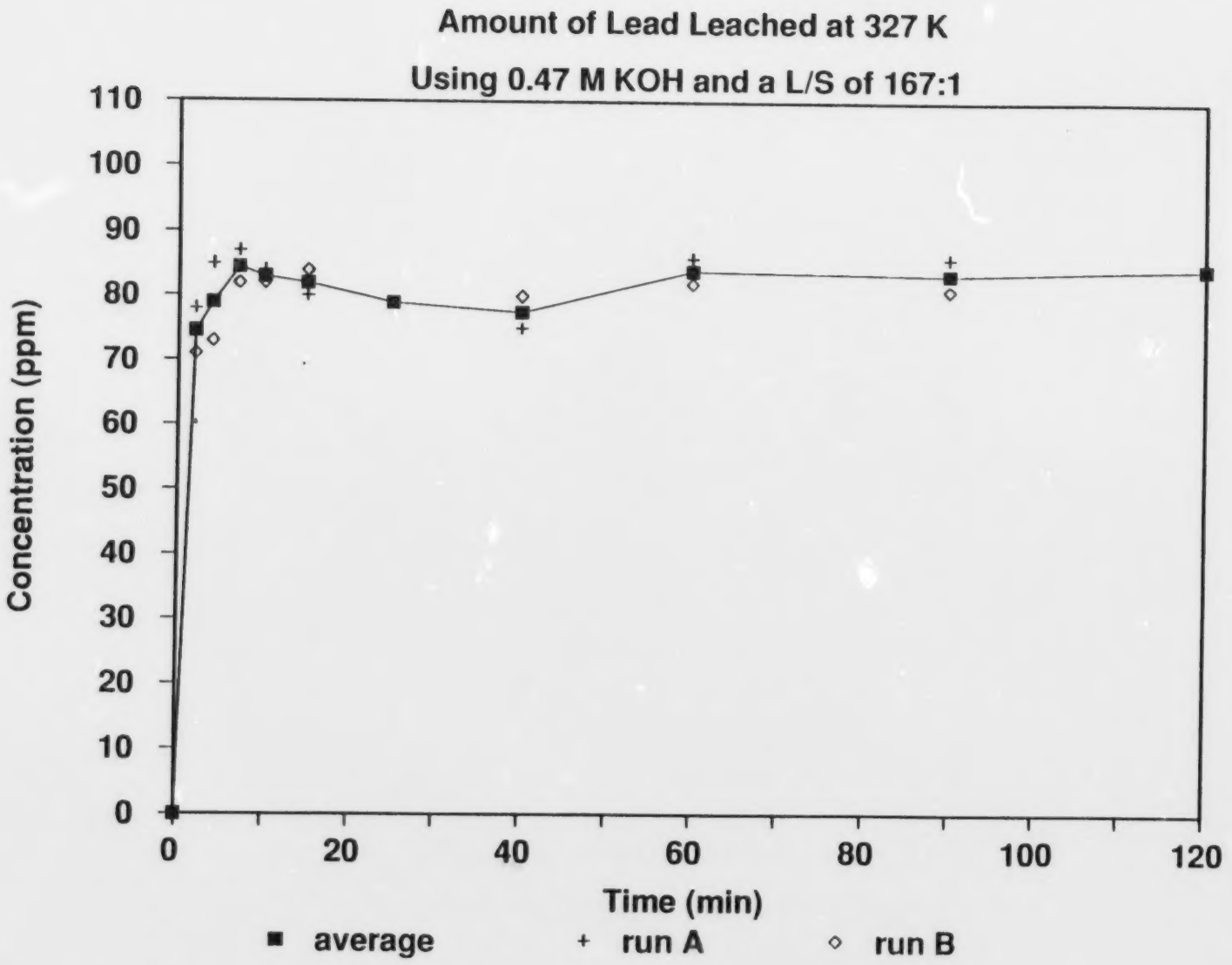

Amount of Lead Leached at a L/S of 20:1
Using 0.47 M KOH
Concentration (ppm)
(Thousands)
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

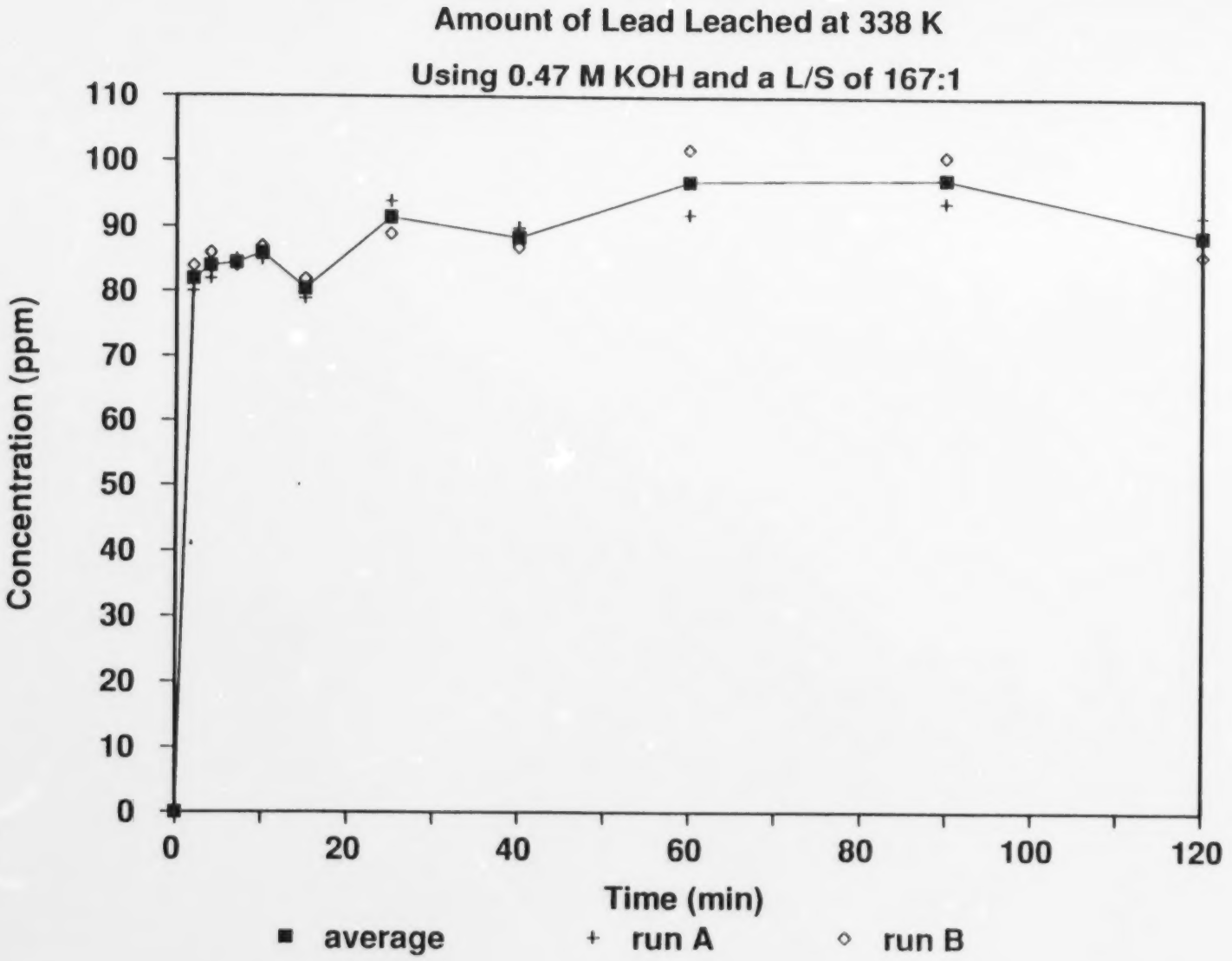

Amount of Lead Leached at a L/S of 10:1
Using 0.47 M KOH
Concentration (ppm)
(Thousands)
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

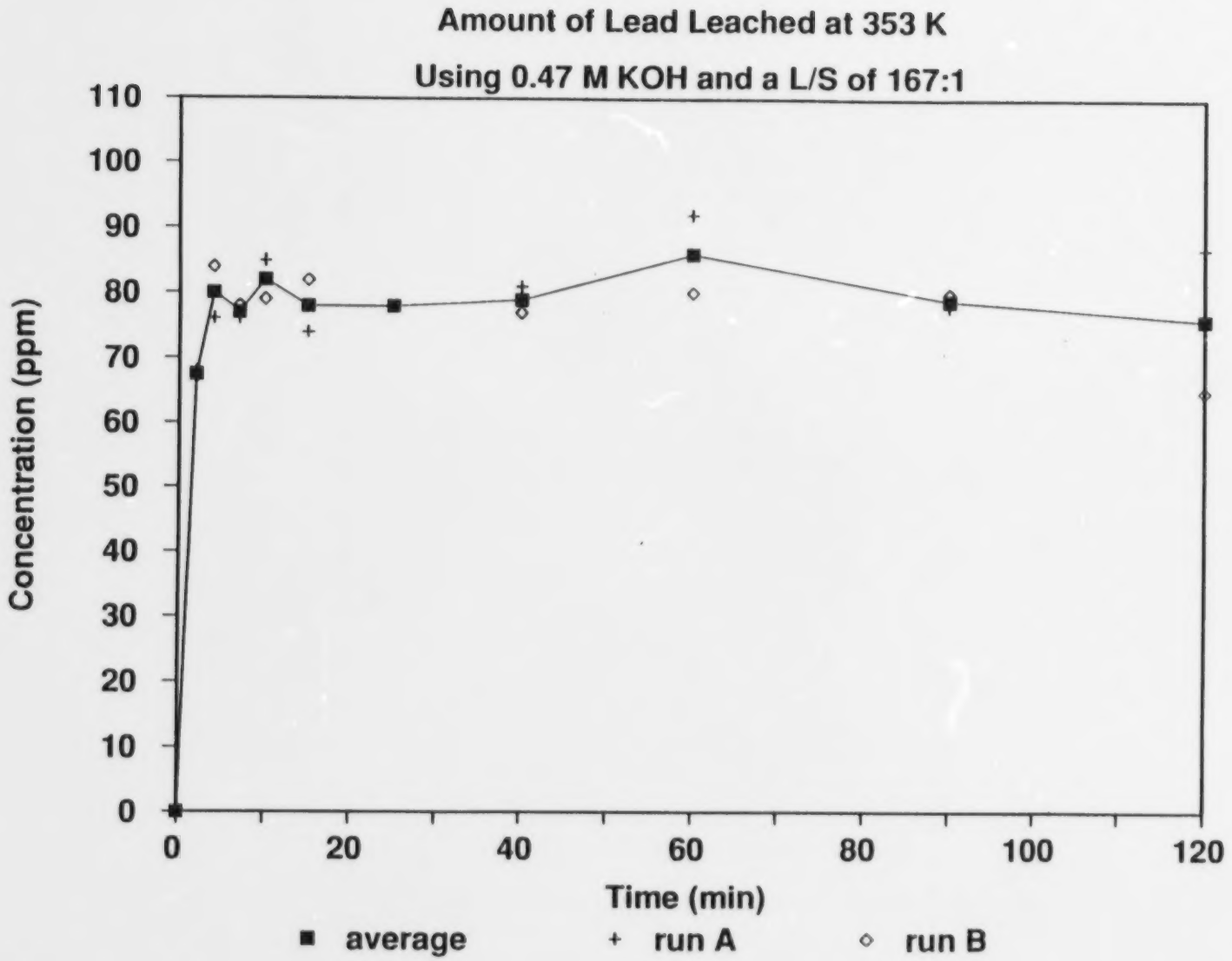

Amount of Lead Leached at a L/S of 5:1
Using 0.47 M KOH
Concentration (ppm)
(Thousands)
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C

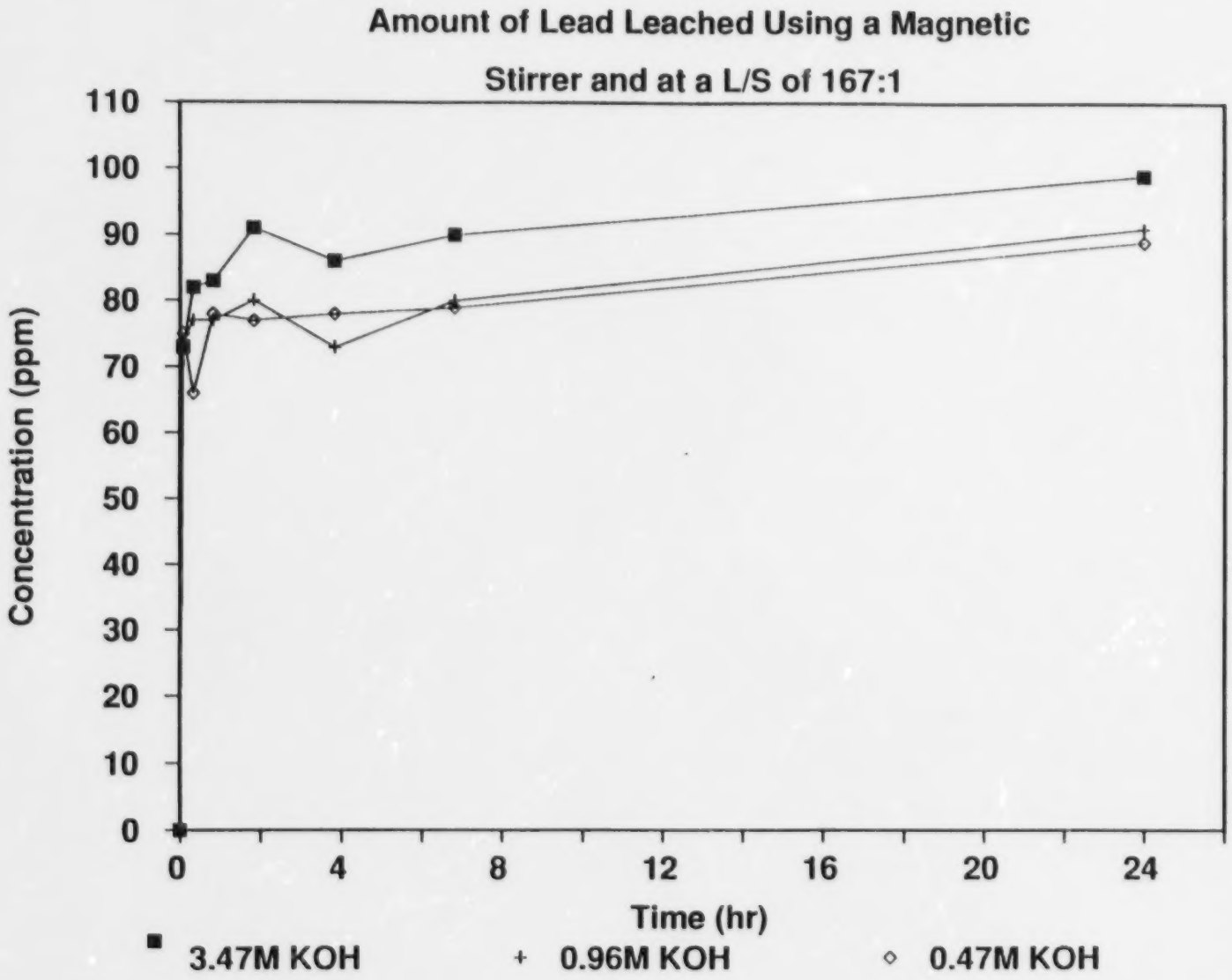

Amount of Lead Leached at a L/S of 3:1
Using 0.47 M KOH
Concentration (ppm)
(Thousands)
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
4
8
12
16
20
24
Time (hr)
average
run A
run B
run C


Amount of Lead Leached at 315 K
Using 0.47 M KOH and a L/S of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Time (min)
average
run A
run B


Amount of Lead Leached at 327 K
Using 0.47 M KOH and a L/S of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Time (min)
average
run A
run B


Amount of Lead Leached at 338 K
Using 0.47 M KOH and a L/S of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Time (min)
average
run A
run B


Amount of Lead Leached at 353 K
Using 0.47 M KOH and a L/S of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
20
40
60
80
100
120
Time (min)
average
run A
run B


Amount of Lead Leached Using a Magnetic Stirrer and at a L/S of 167:1
Concentration (ppm)
110
100
90
80
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
Time (hr)
3.47M KOH
0.96M KOH
0.47M KOH